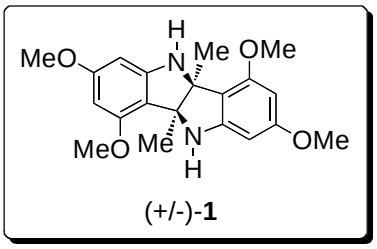
Bisindolines from the Reaction of 3,5-Dimethoxyaniline with Vicinal Diones

*James Kovach, William W. Brennessel, and William D. Jones**

Department of Chemistry, University of Rochester, Rochester, New York 14627

Supporting Information

Table of Contents



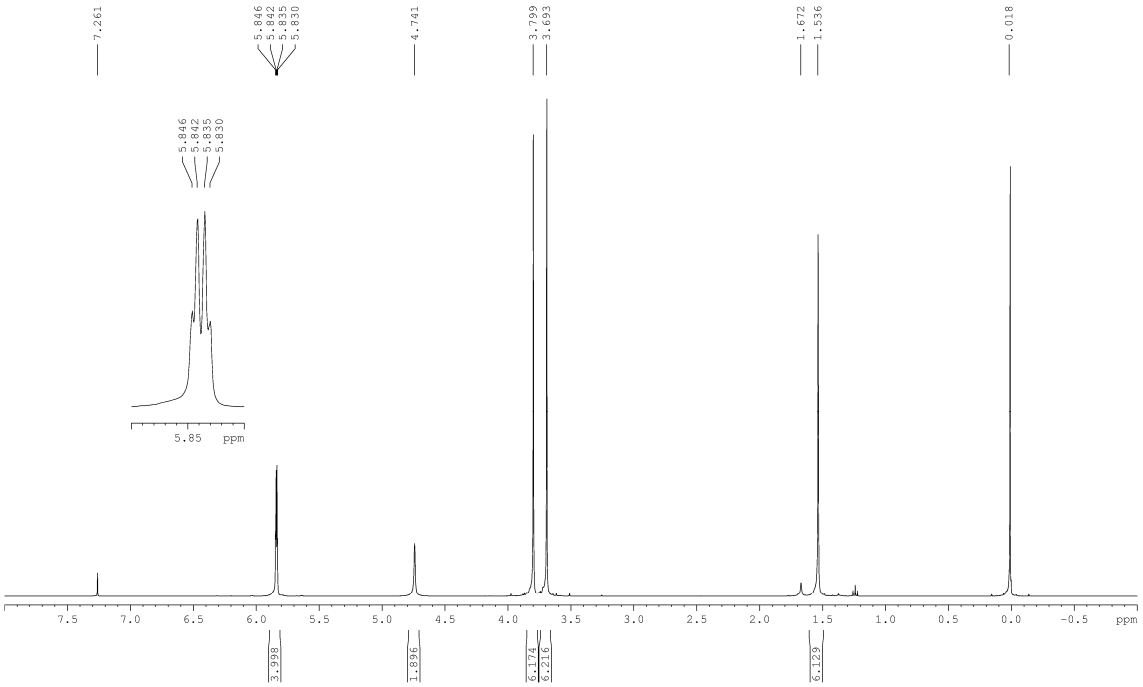
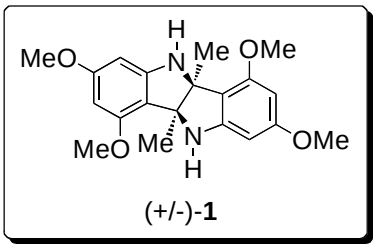


Figure S1. ^{1}H NMR spectrum of (+/-)-**1** (400 MHz, $CDCl_3$, 25 °C).



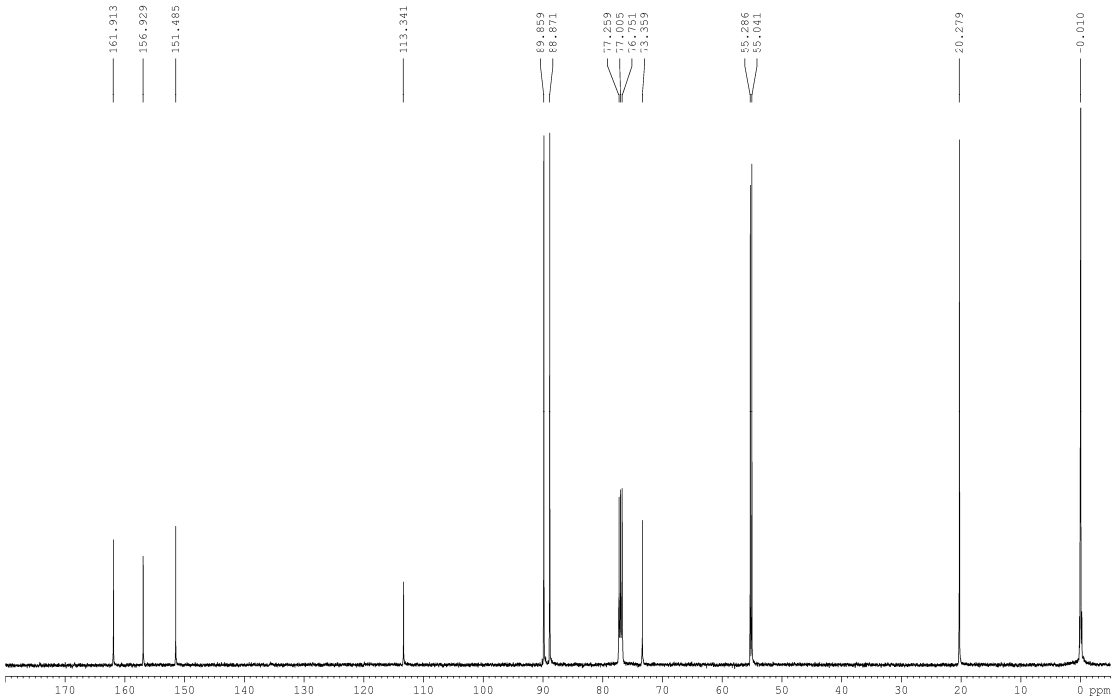
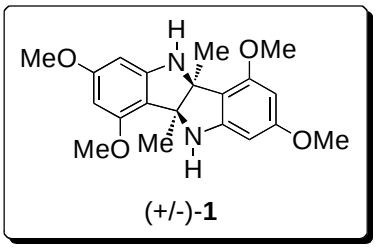


Figure S2. $^{13}C\{^{1}H\}$ NMR spectrum of (+/-)-**1** (125 MHz, $CDCl_3$, 25 ºC).



(+/-)-**1**

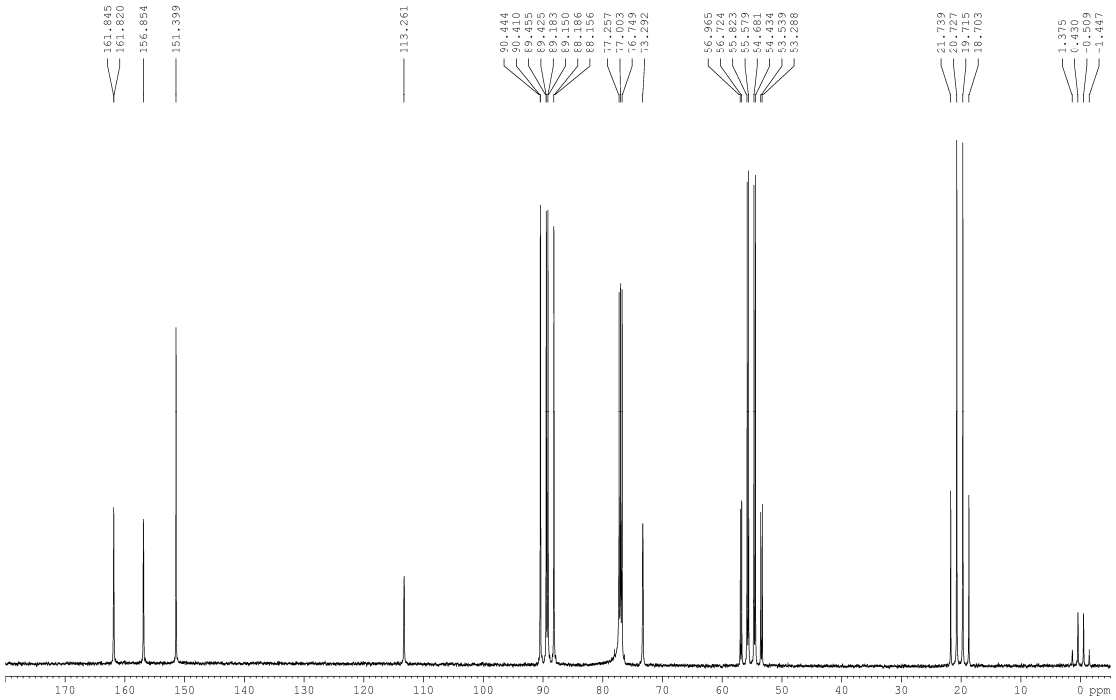
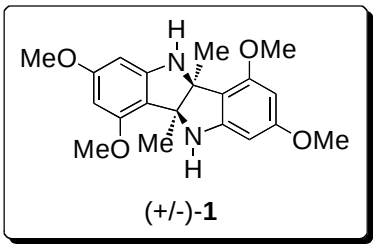


Figure S3. ^{13}C NMR spectrum of (+/-)-**1** (125 MHz, $CDCl_3$, 25 ºC).



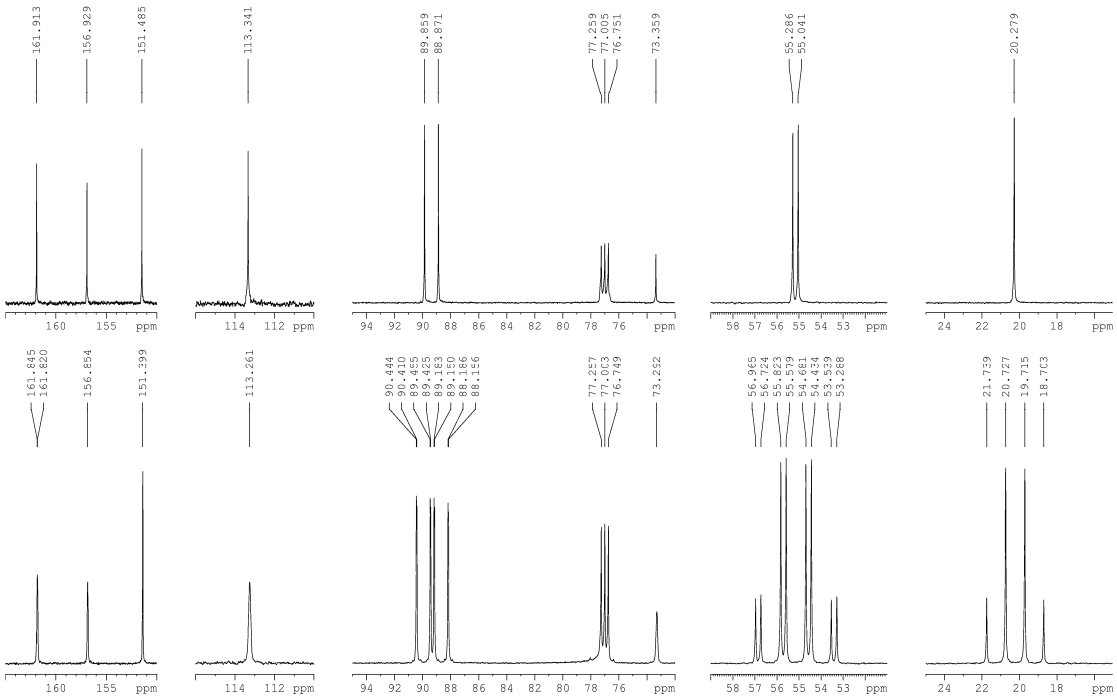


Figure S4. $^{13}C\{^{1}H\}$ and ^{13}C NMR spectra overlay of (+/-)-**1** (125 MHz, $CDCl_3$, 25 ºC). Peak heights are normalized to emphasize splitting.

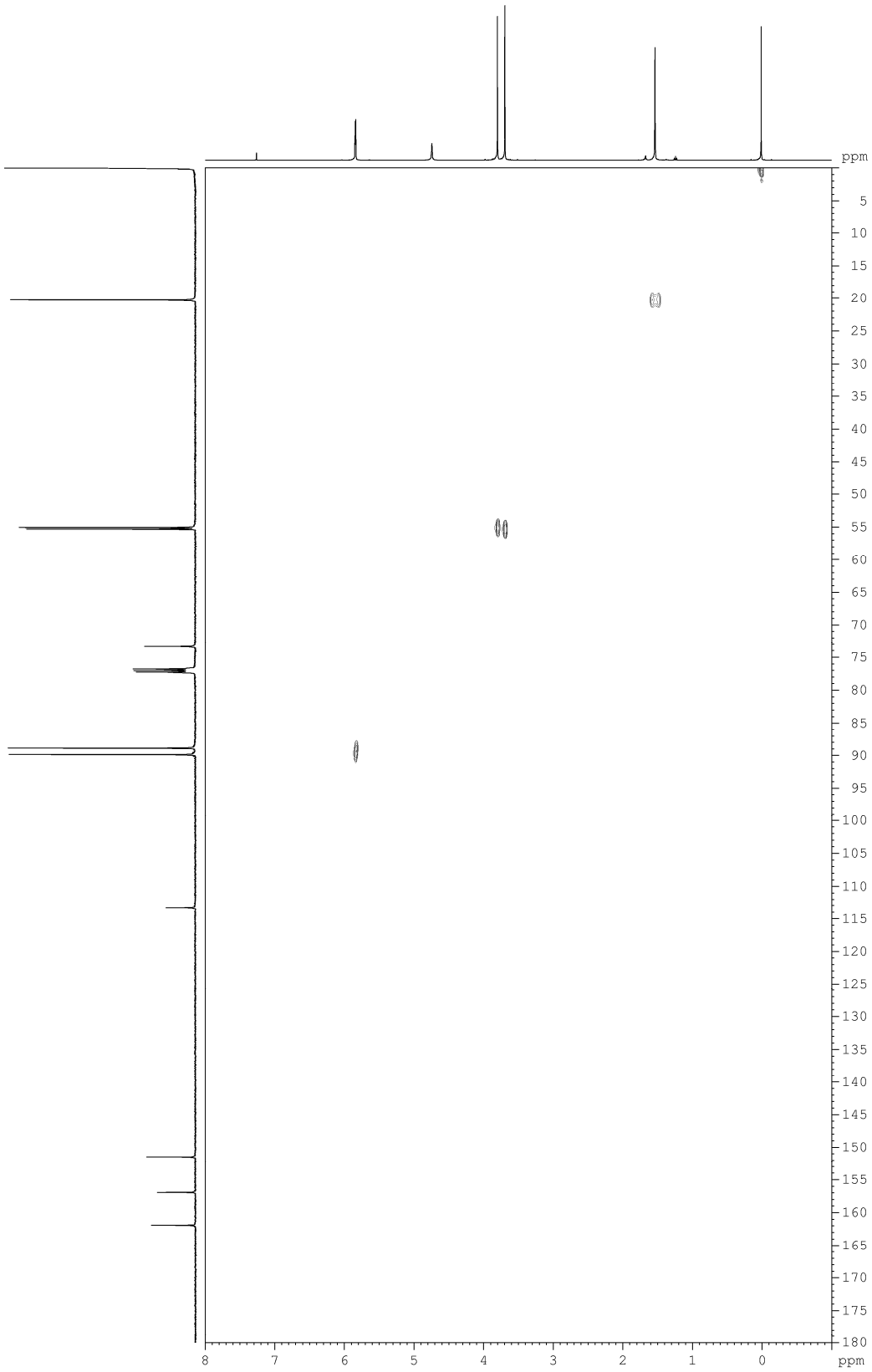


Figure S5. ^{1}H-^{13}C HSQC NMR spectrum of (+/-)-**1** (125 MHz, $CDCl_3$, 25 ºC).

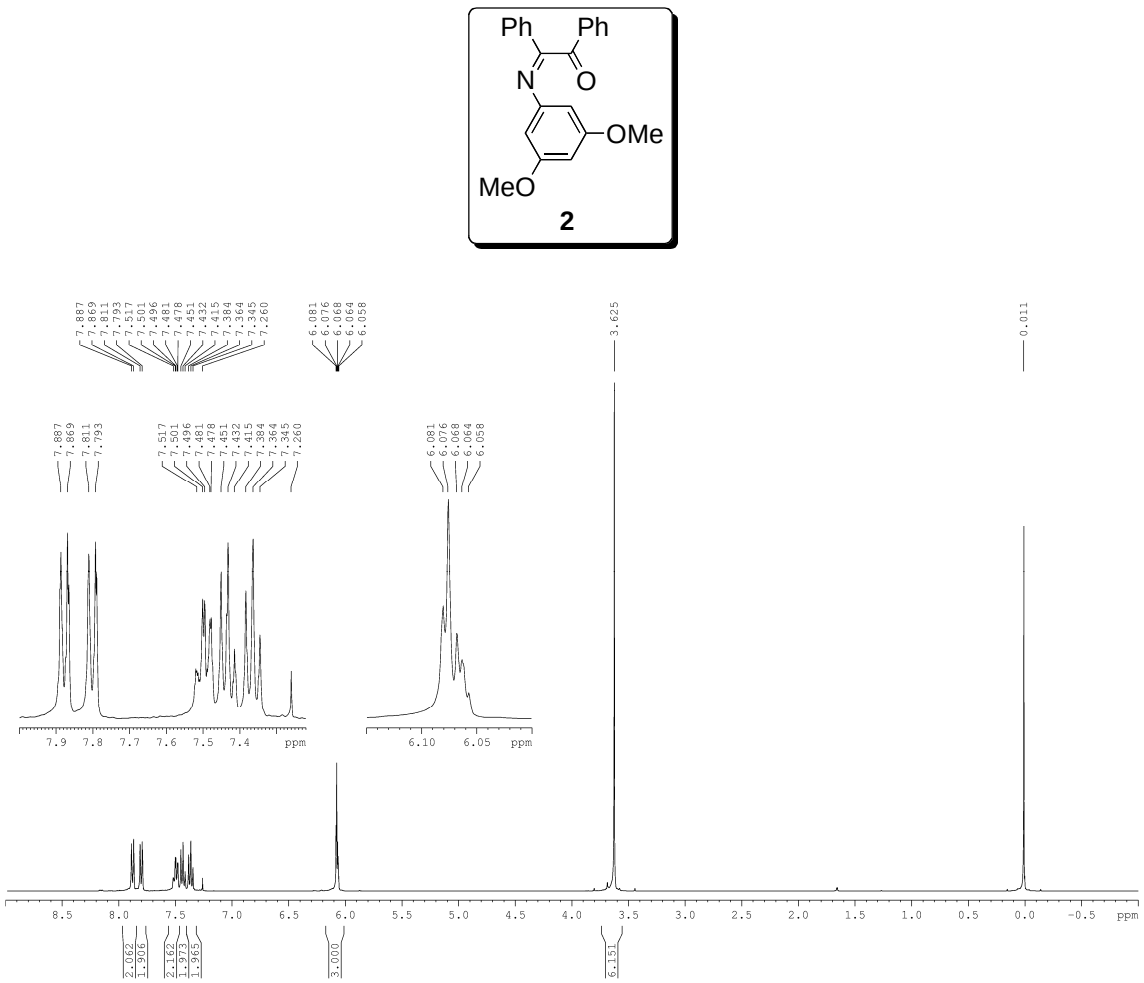
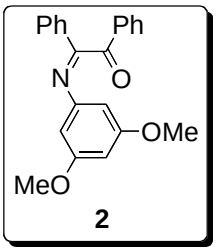


Figure S6. ^{1}H NMR spectrum of **2** (400 MHz, $CDCl_3$, 25 °C).



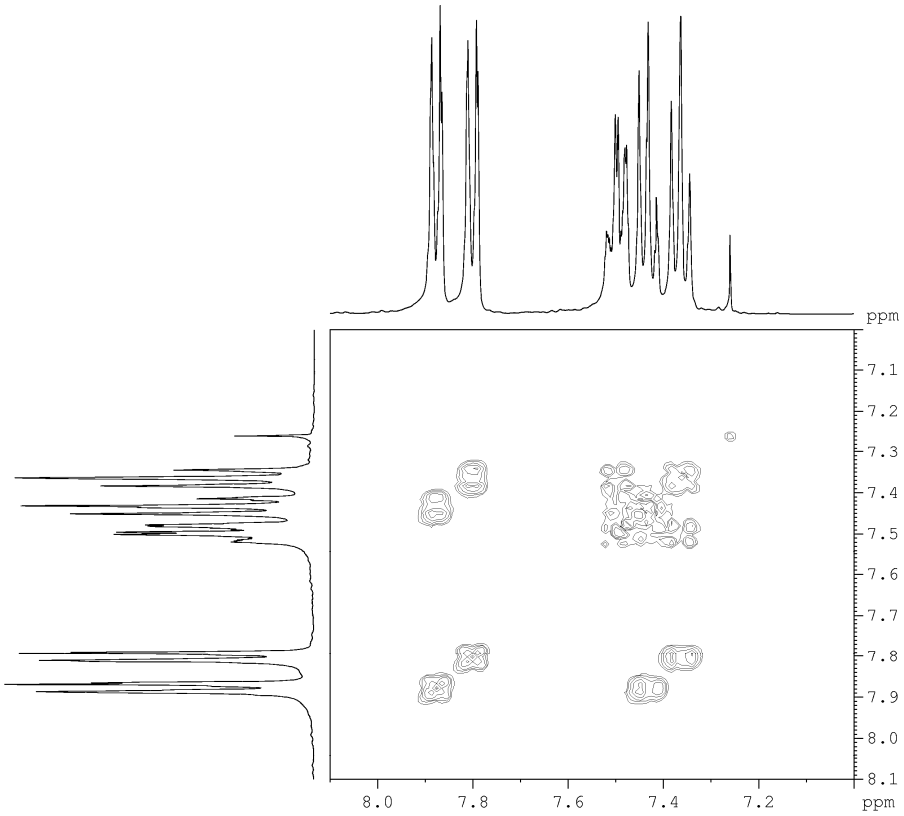


Figure S7. ^{1}H-^{1}H COSY NMR spectrum of **2** zoomed in on the aromatic region (400 MHz, $CDCl_3$, 25 ºC).

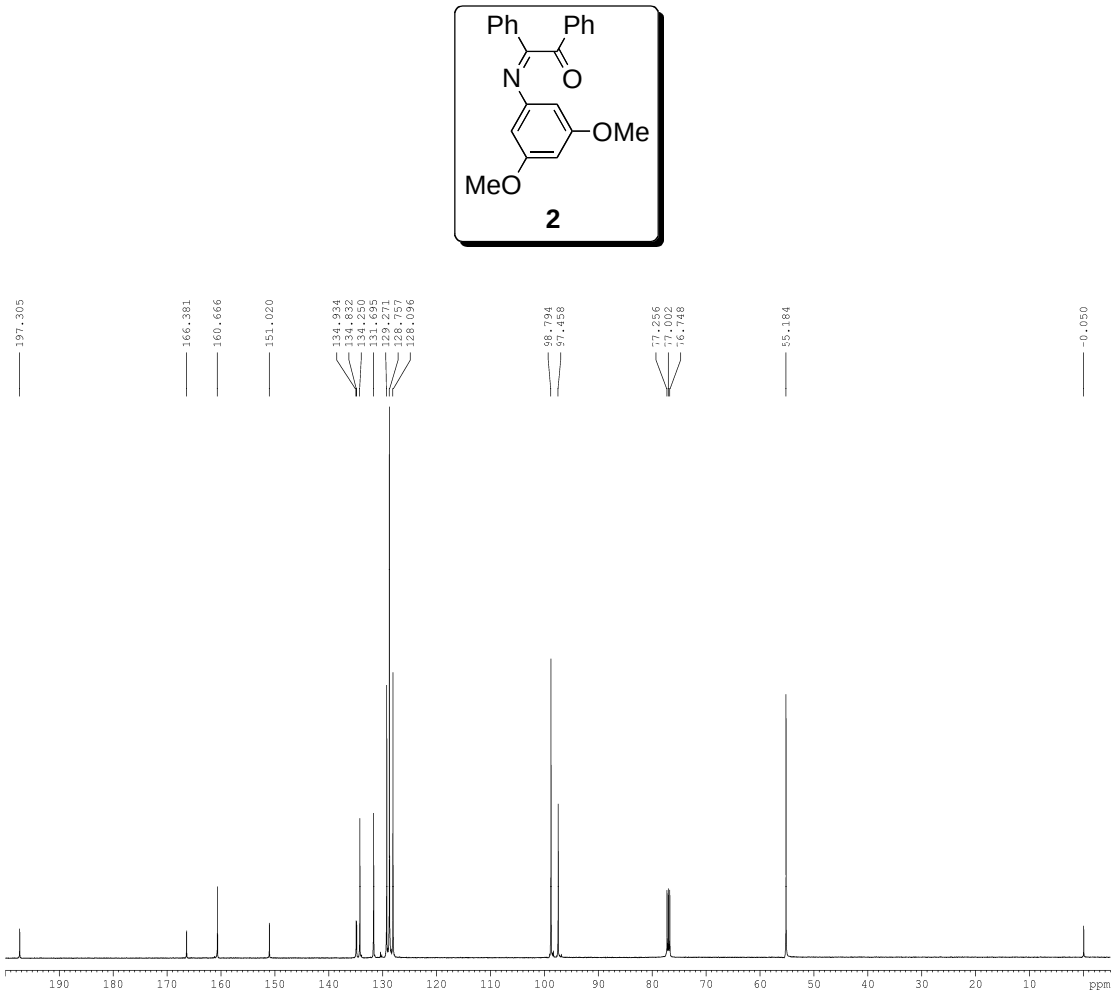


Figure S8. $^{13}C\{^{1}H\}$ NMR spectrum of **2** (125 MHz, $CDCl_3$, 25 ºC).

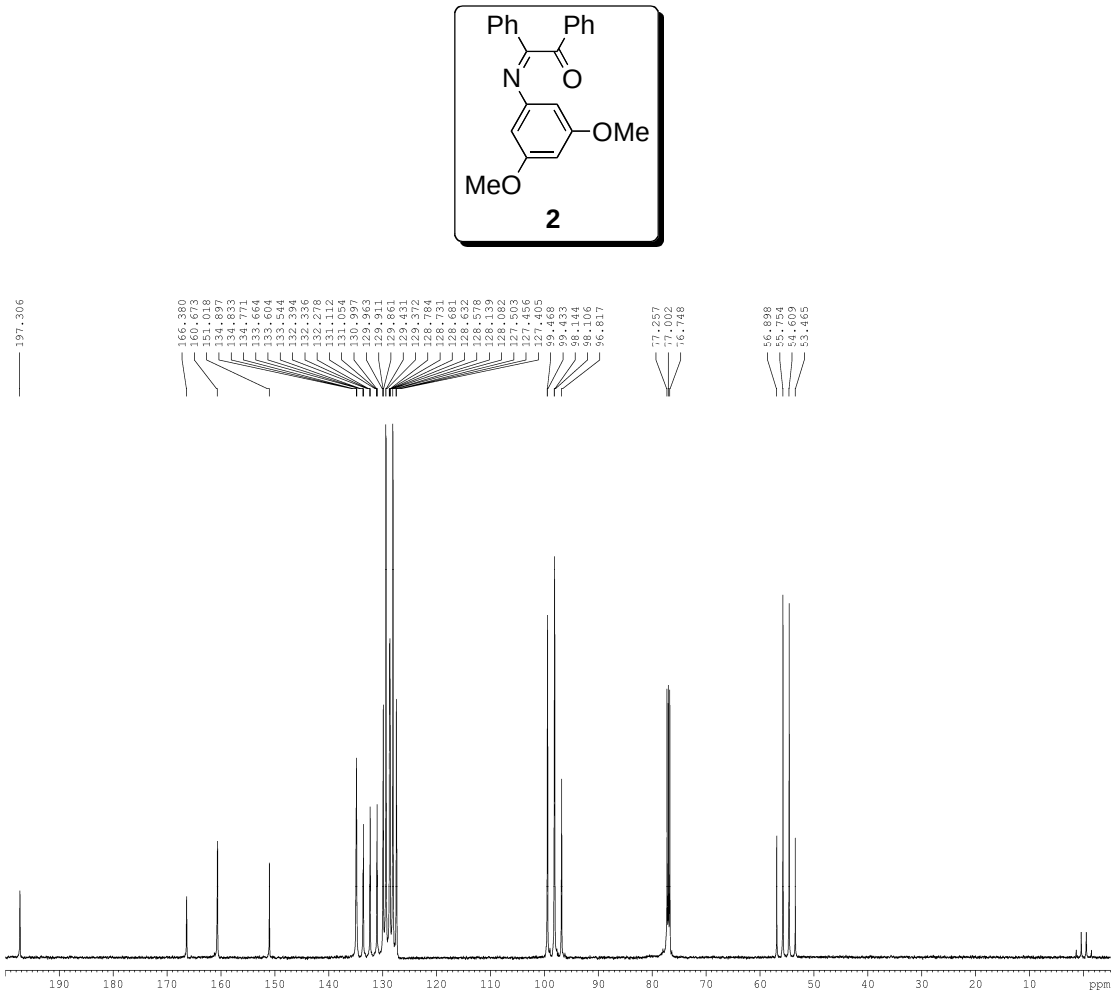


Figure S9. ^{13}C NMR spectrum of **2** (125 MHz, $CDCl_3$, 25 ºC).

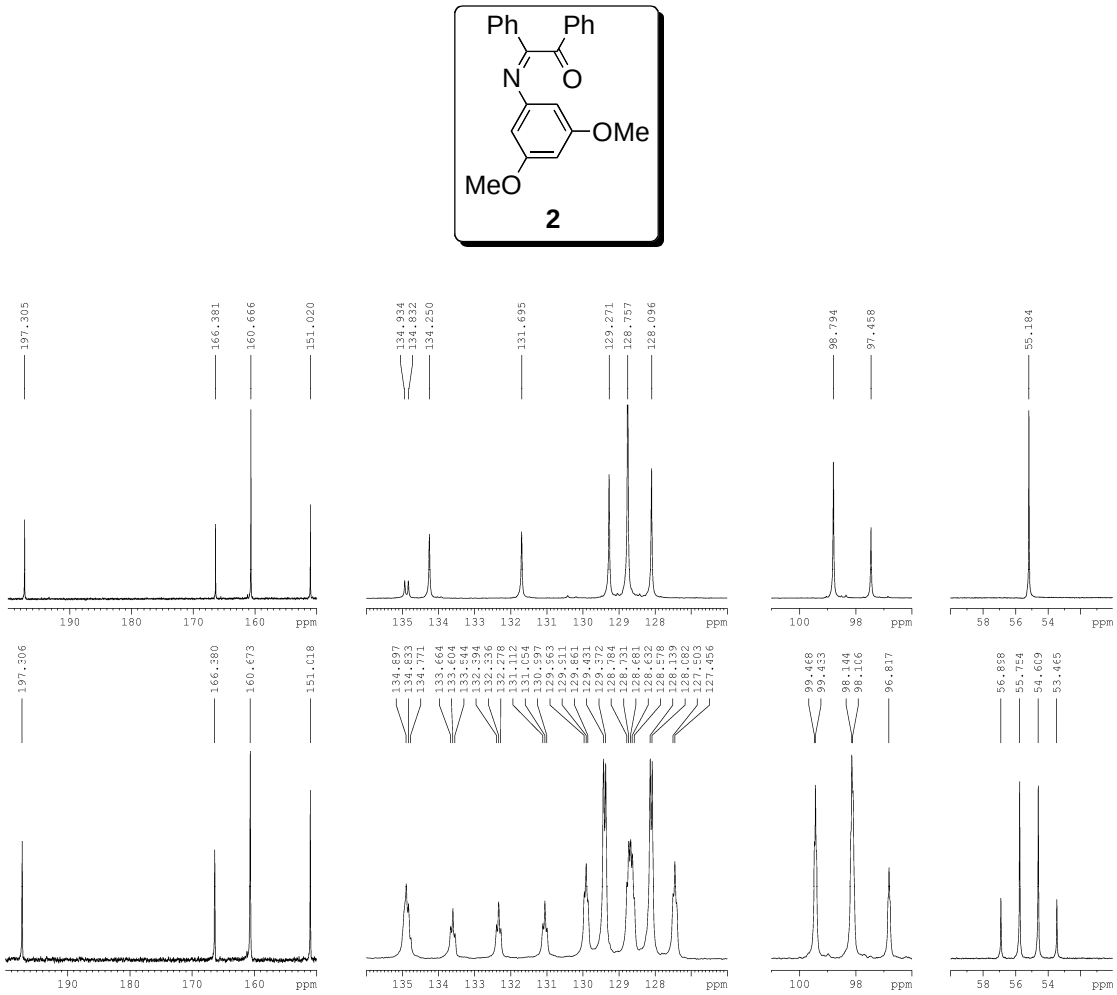


Figure S10. $^{13}C\{^{1}H\}$ and ^{13}C NMR spectra overlay of **2** (125 MHz, $CDCl_3$, 25 ºC). Peak heights are normalized to emphasize splitting.

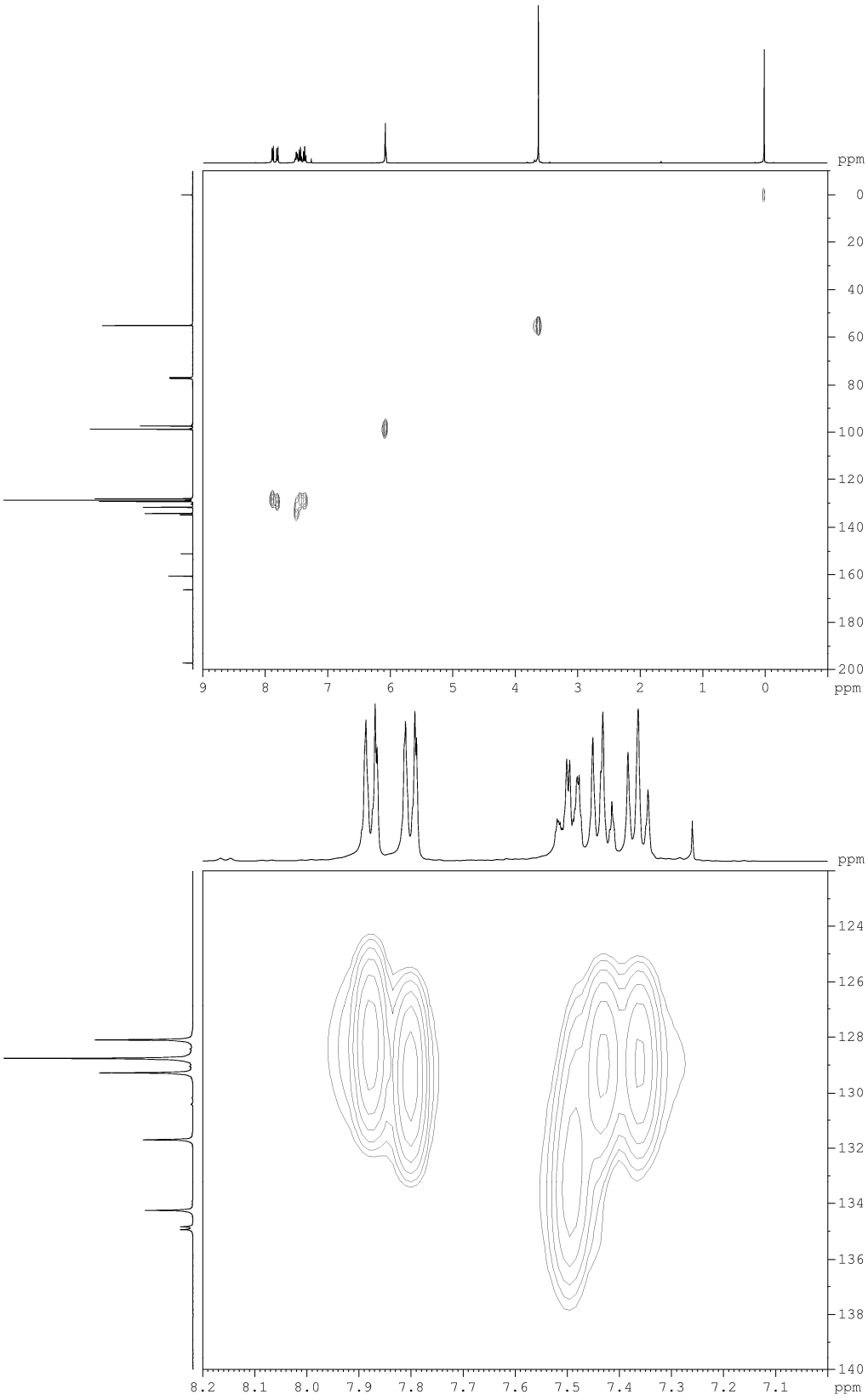


Figure S11. ^{1}H-^{13}C HSQC NMR spectrum of **2** (125 MHz, $CDCl_3$, 25 ºC).

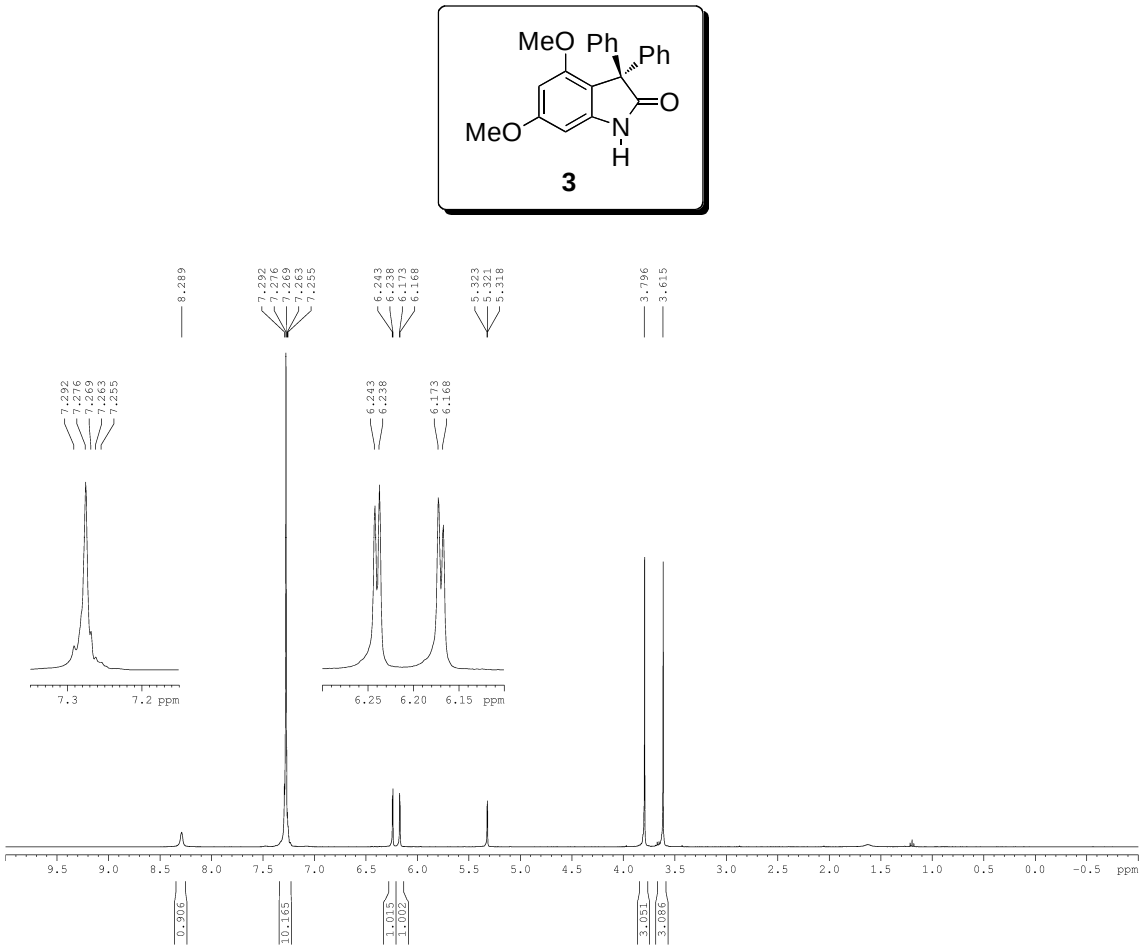


Figure S12. ^{1}H NMR spectrum of **3** (400 MHz, CD_2Cl_2, 25 °C).

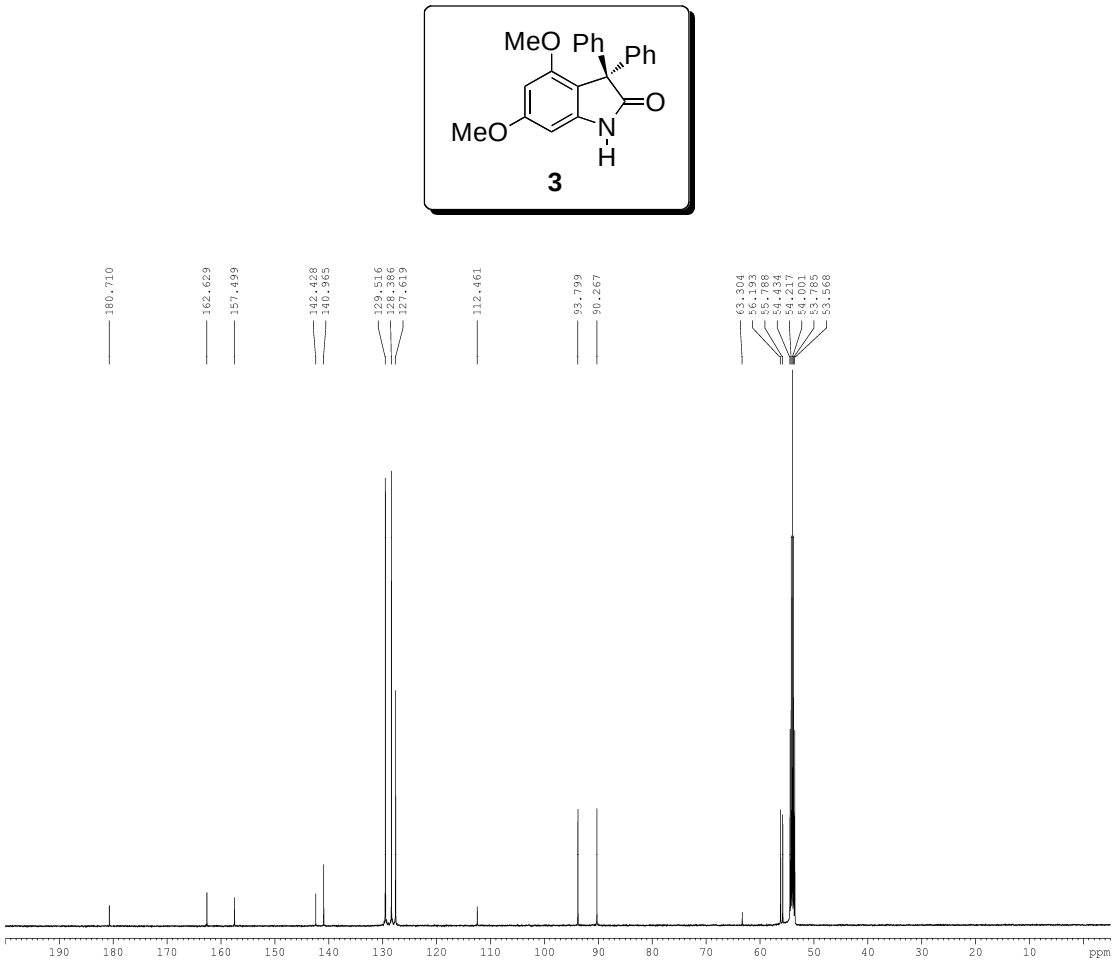


Figure S13. $^{13}C\{^{1}H\}$ NMR spectrum of **3** (125 MHz, CD_2Cl_2, 25 ºC).

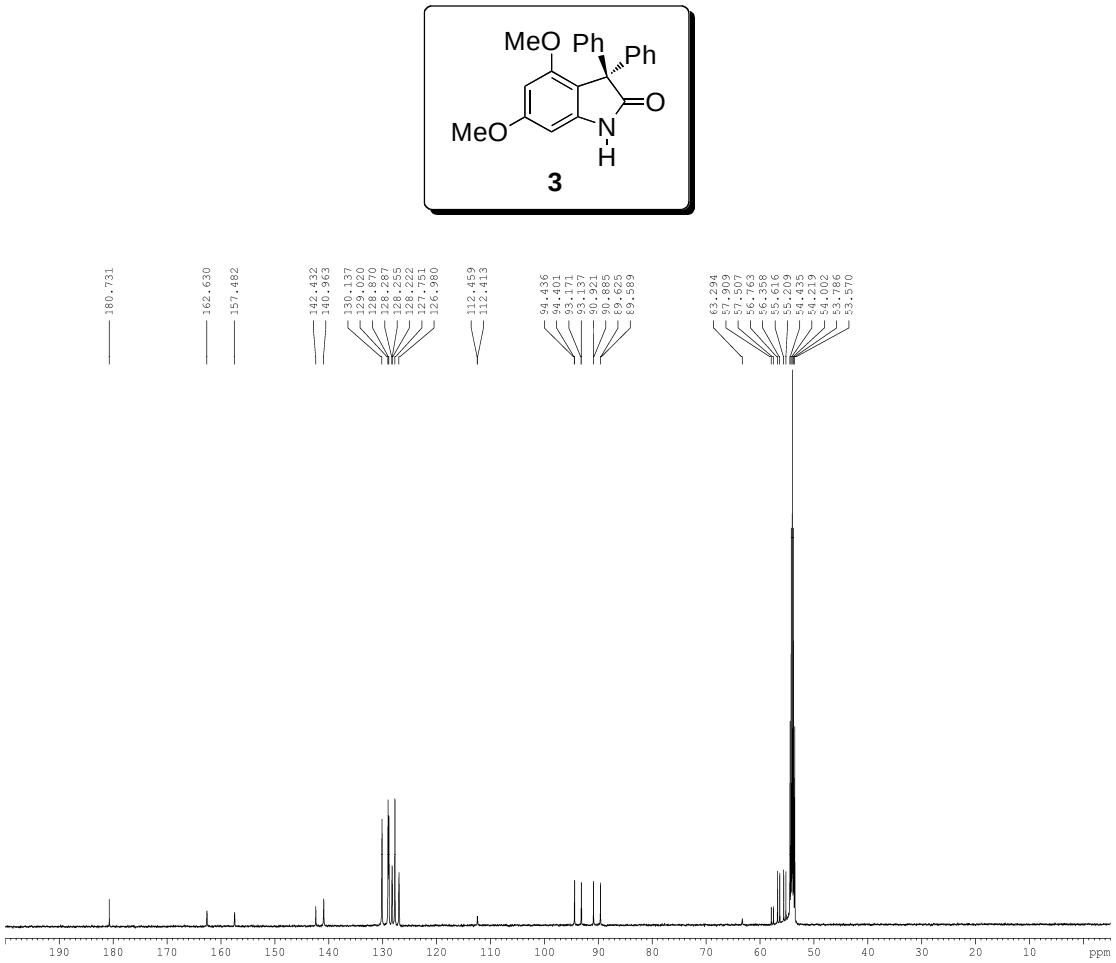
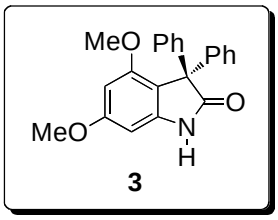


Figure S14. ^{13}C NMR spectrum of **3** (125 MHz, CD_2Cl_2, 25 ºC).



3

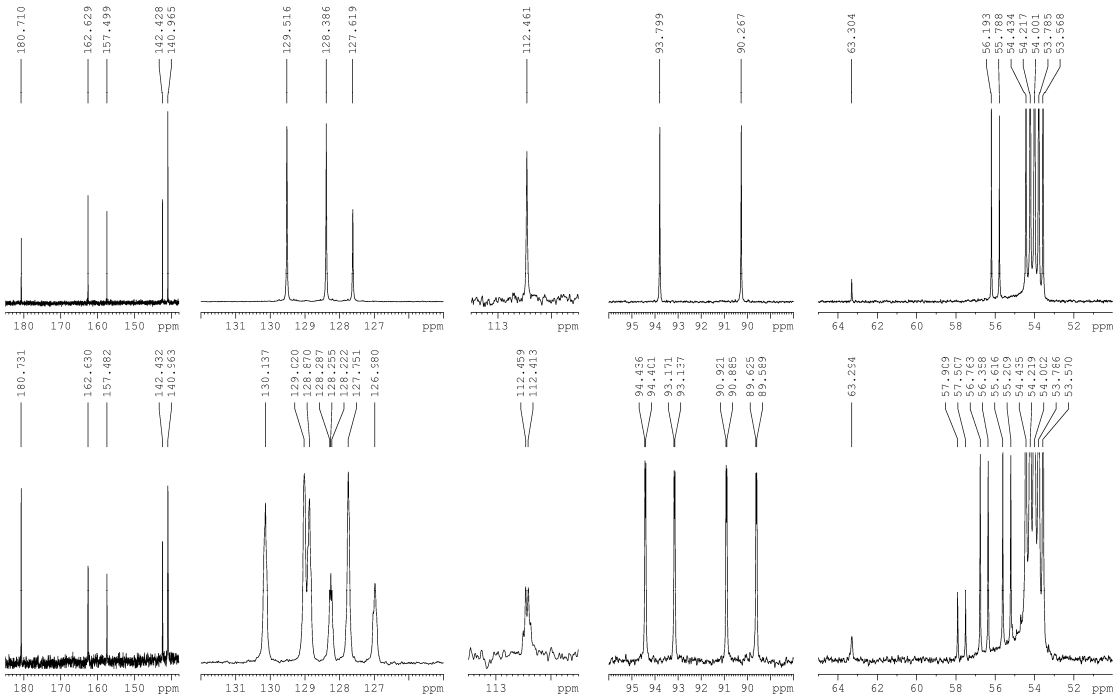


Figure S15. $^{13}C\{^{1}H\}$ and ^{13}C NMR spectra overlay of **3** (125 MHz, CD_2Cl_2, 25 ºC). Peak heights are normalized to emphasize splitting.

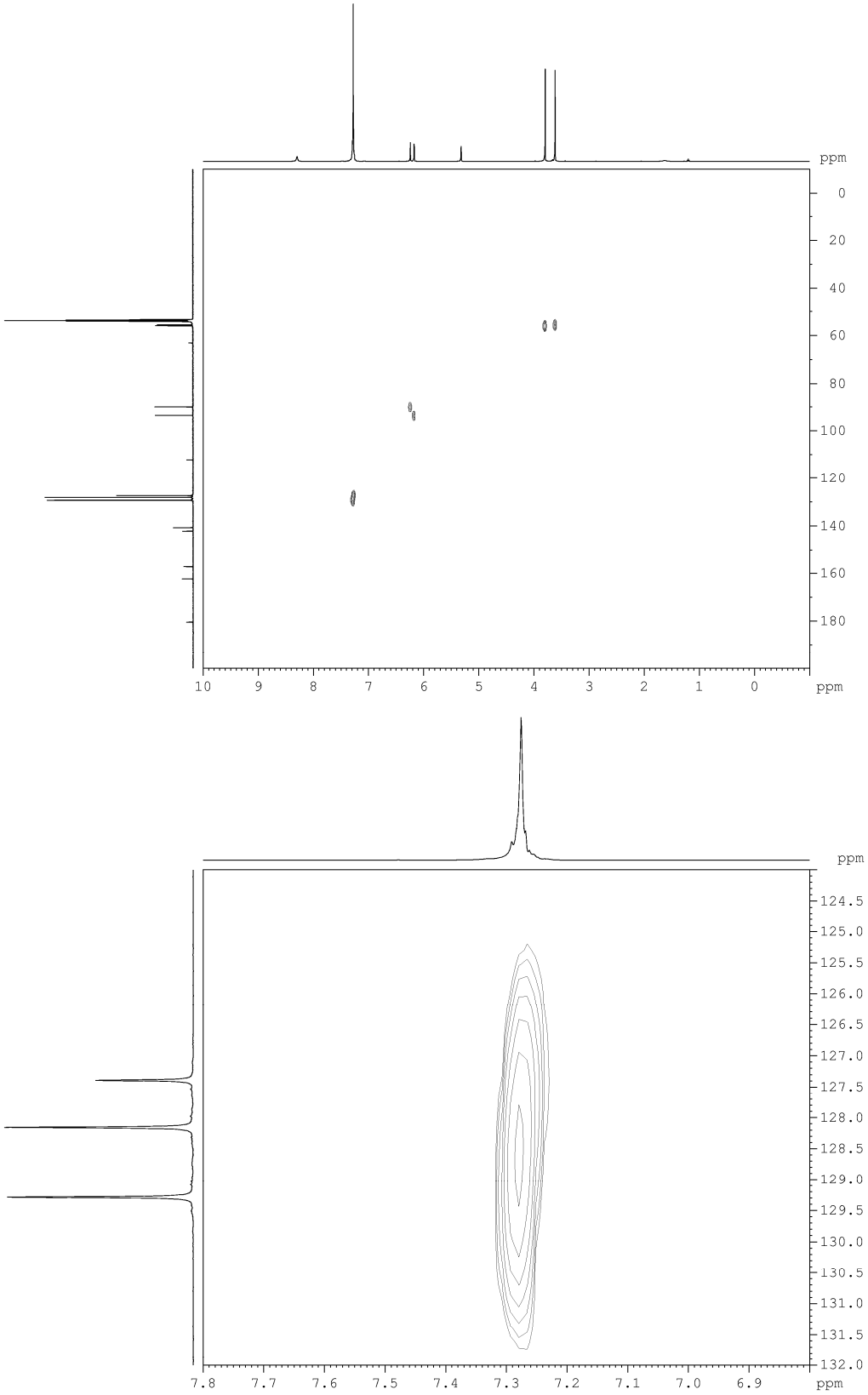
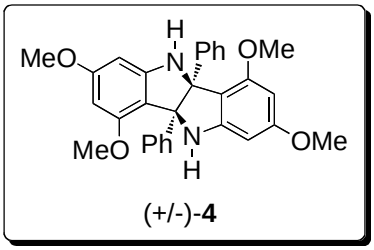


Figure S16. ^{1}H-^{13}C HSQC NMR spectrum of **3** (125 MHz, CD_2Cl_2, 25 ºC).



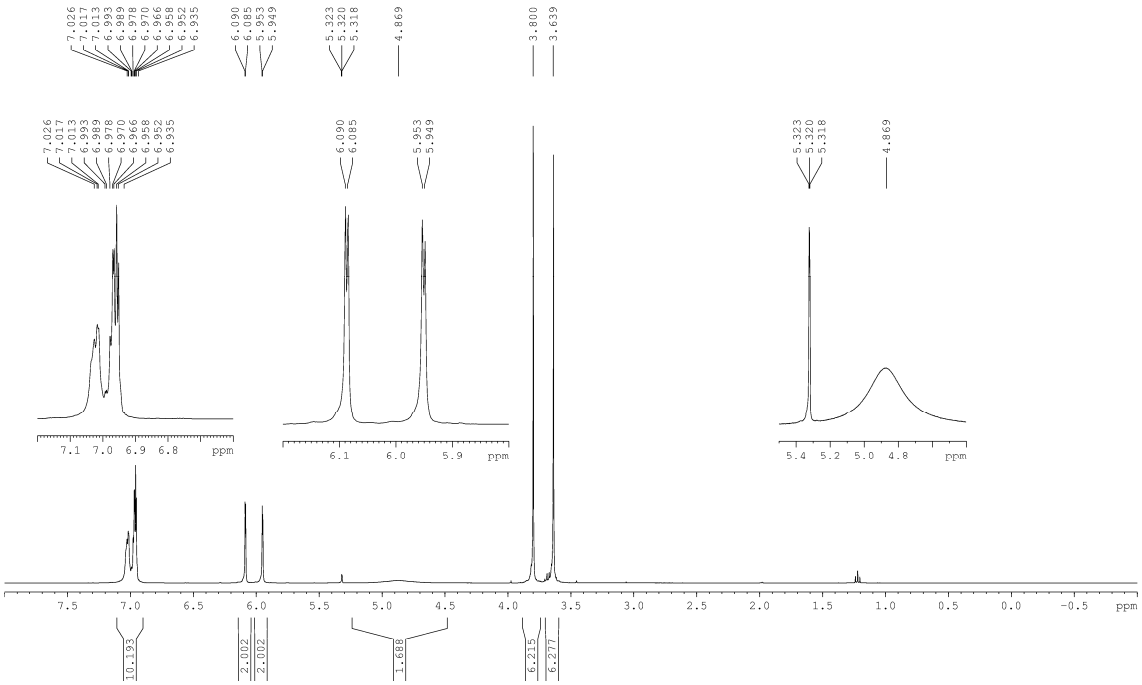


Figure S17. ^{1}H NMR spectrum of (+/-)-**4** (400 MHz, CD_2Cl_2, 25 °C).

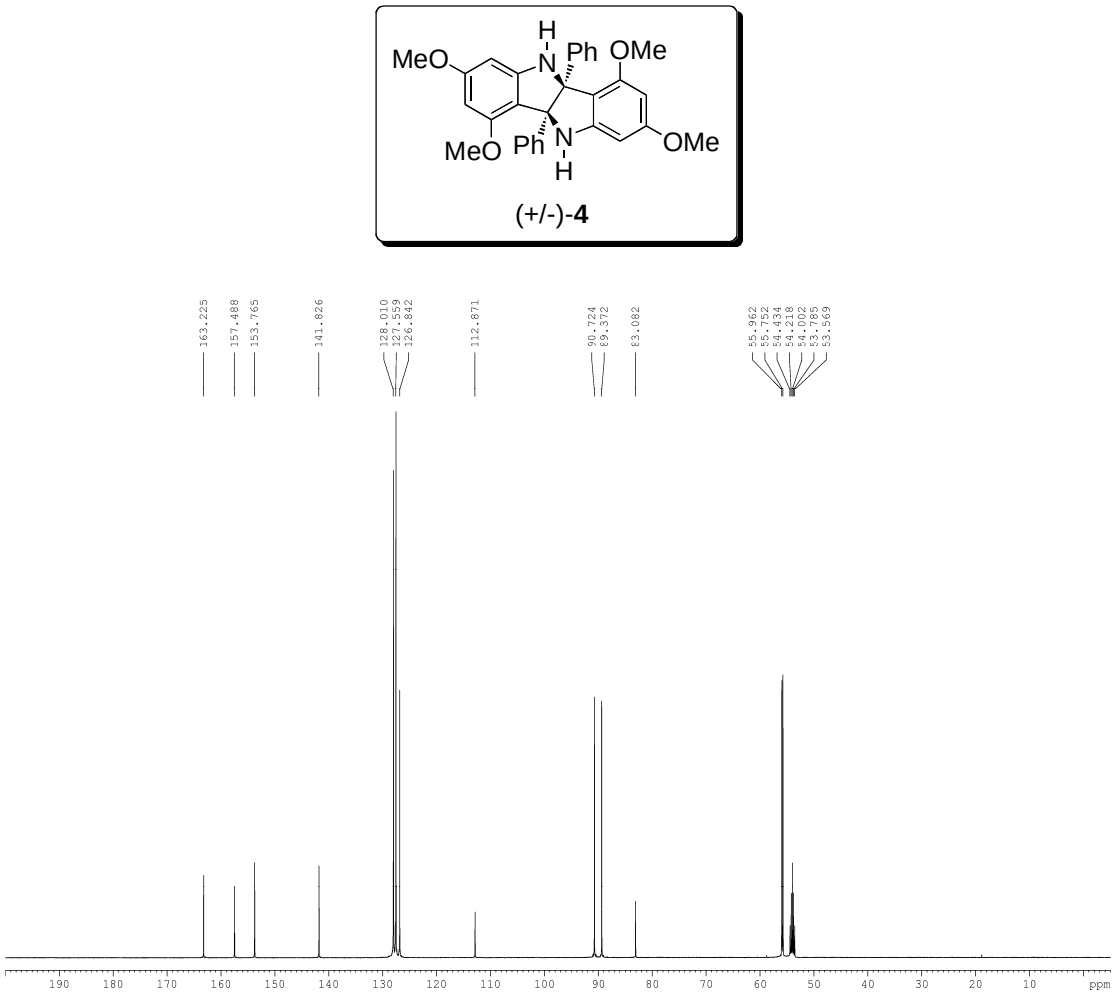


Figure S18. $^{13}C\{^{1}H\}$ NMR spectrum of (+/-)-**4** (125 MHz, CD_2Cl_2, 25 ºC).

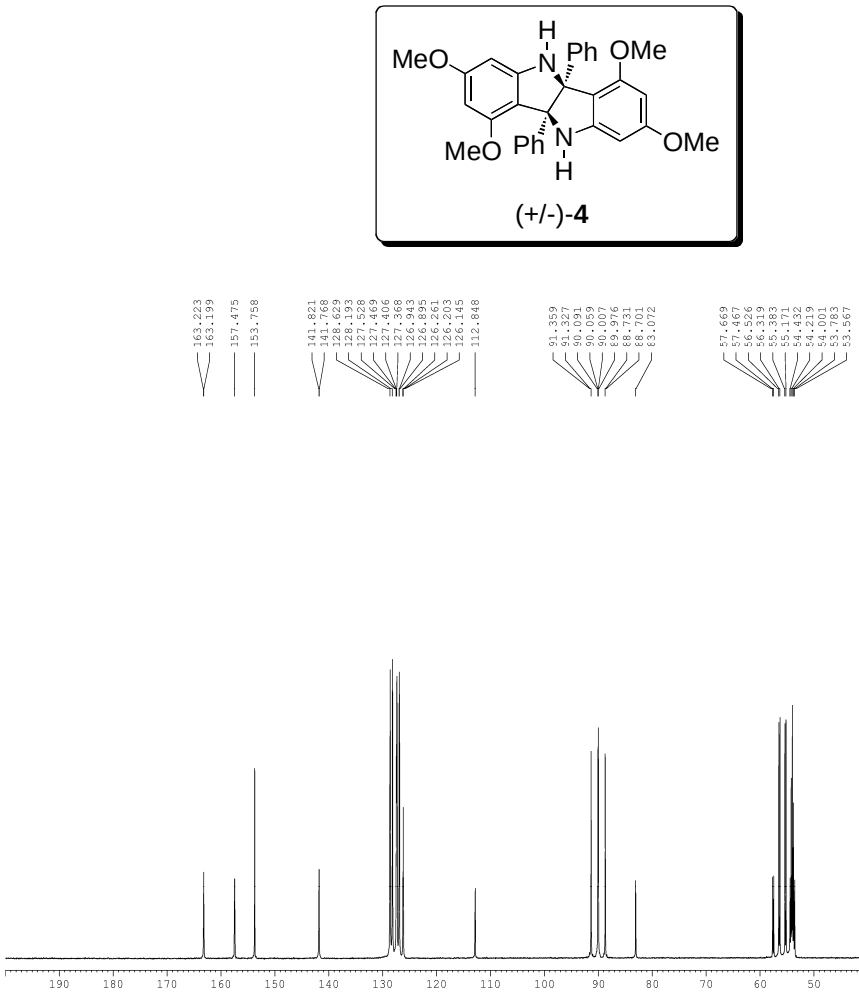
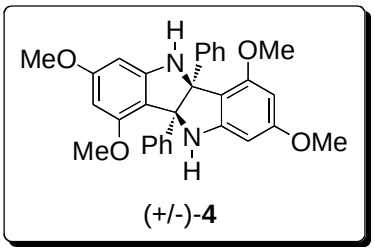


Figure S19. ^{13}C NMR spectrum of (+/-)-**4** (125 MHz, CD_2Cl_2, 25 ºC).



(+/-)-**4**

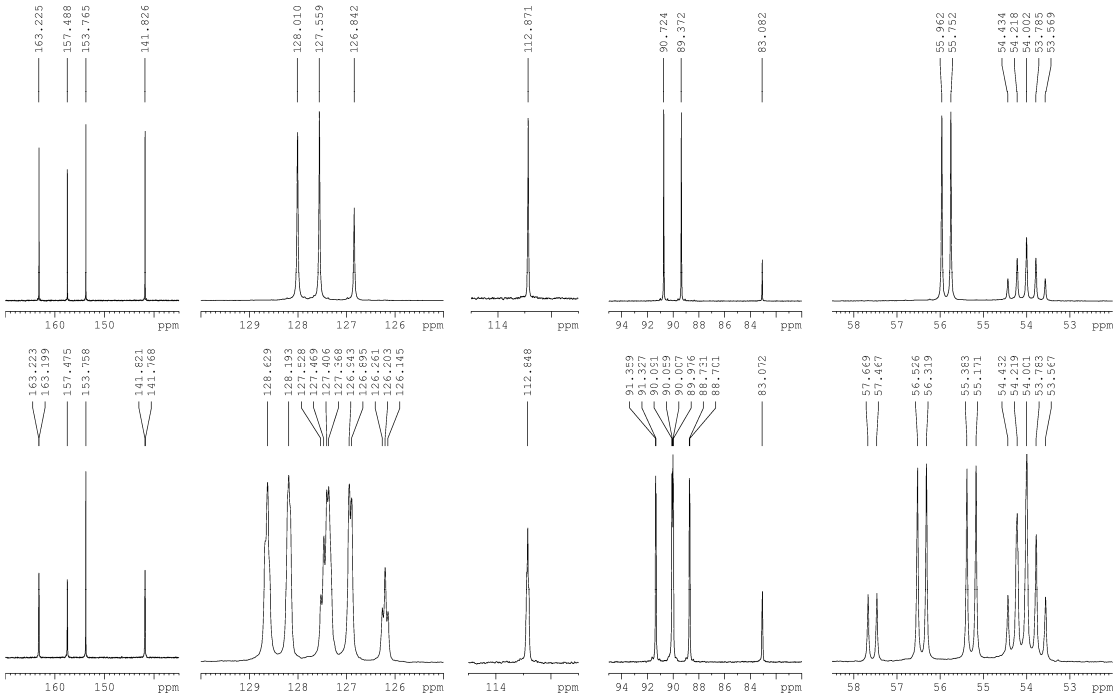


Figure S20. $^{13}C\{^{1}H\}$ and ^{13}C NMR spectra overlay of (+/-)-**4** (125 MHz, CD_2Cl_2, 25 ºC). Peak heights are normalized to emphasize splitting.

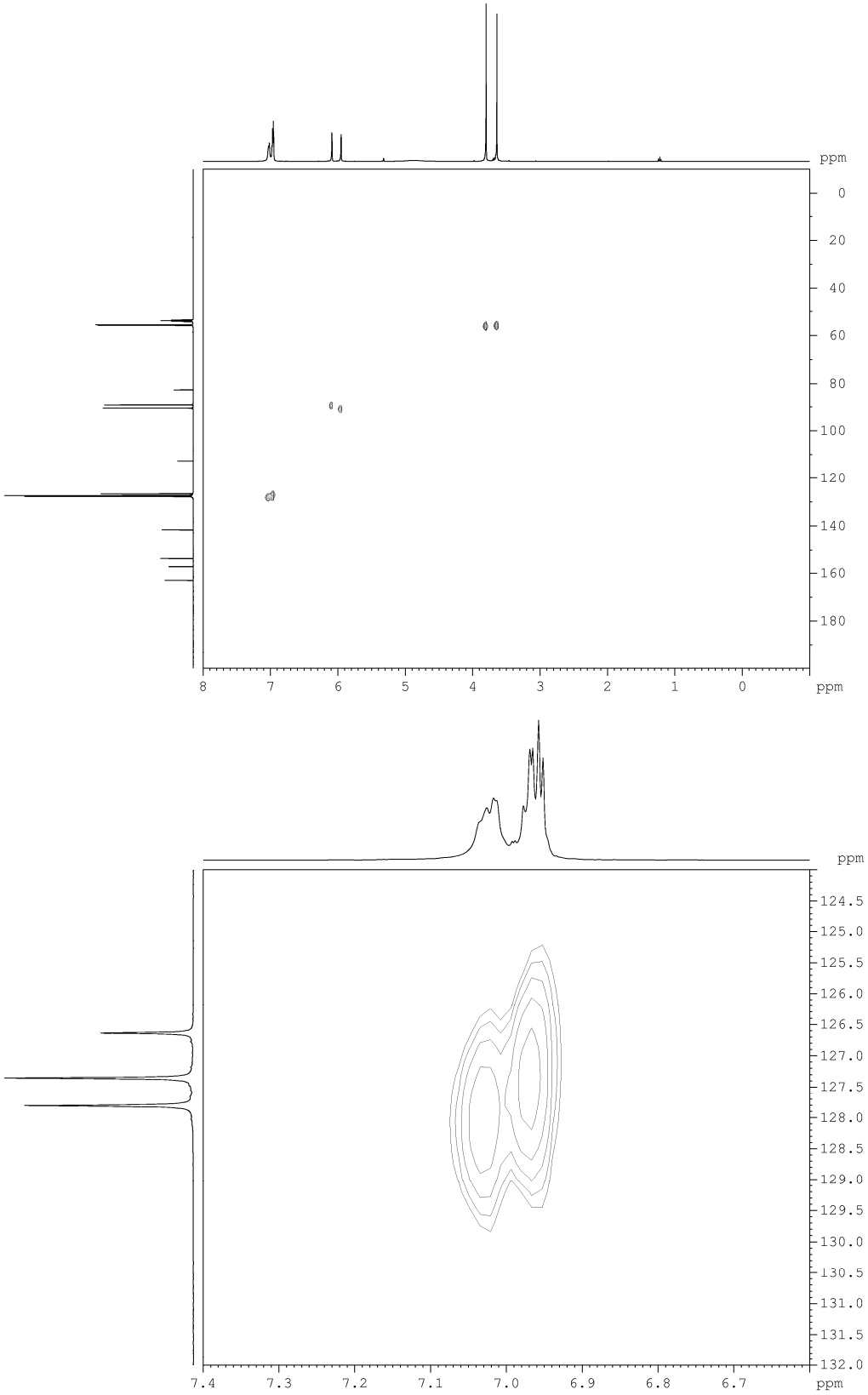


Figure S21. ^{1}H-^{13}C HSQC NMR spectrum of (+/-)-**4** (125 MHz, CD_2Cl_2, 25 ºC).

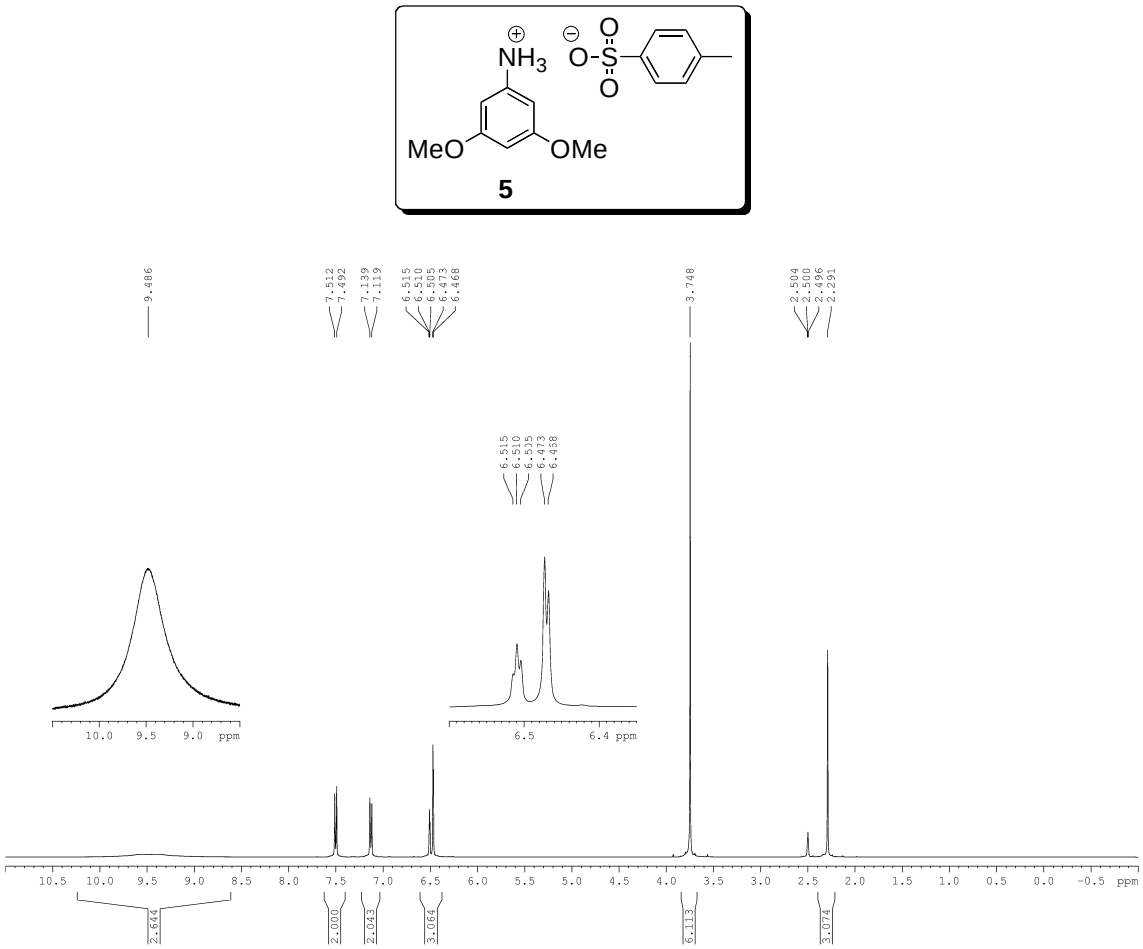
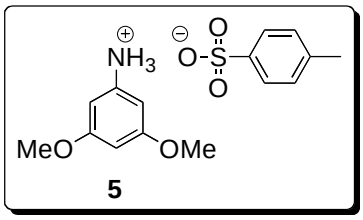


Figure S22. ^{1}H NMR spectrum of **5** (400 MHz, DMSO-d_6, 25 °C).



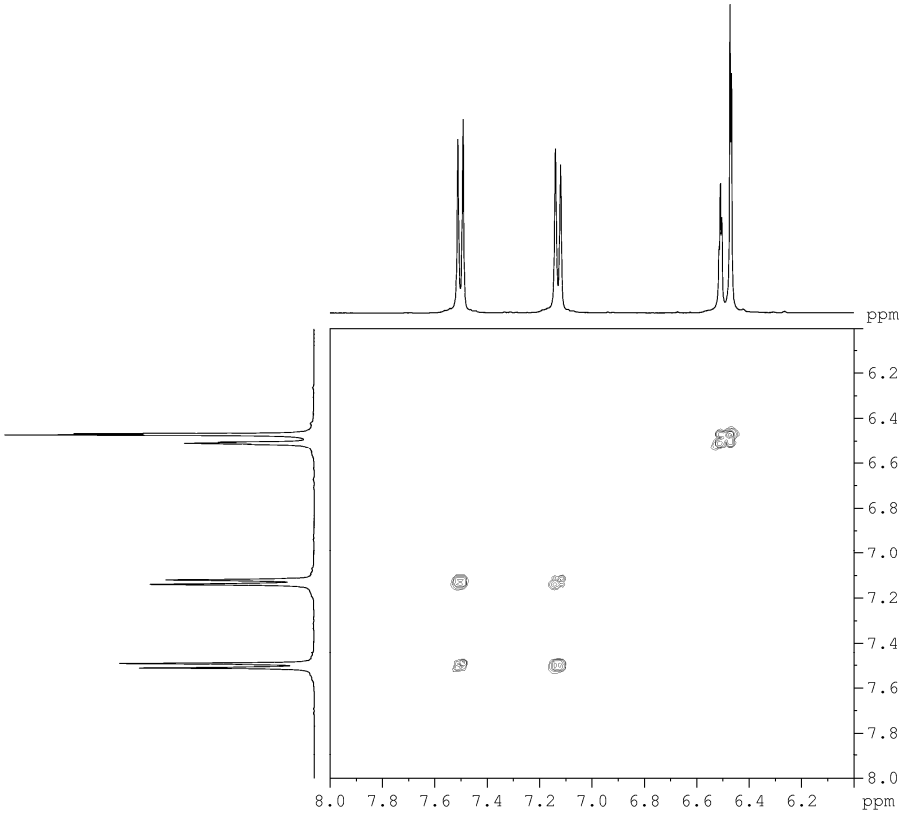
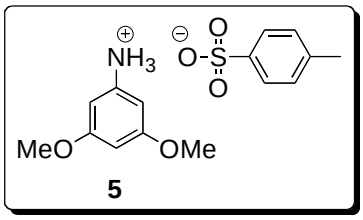


Figure S23. ^{1}H-^{1}H COSY NMR spectrum of **5** zoomed in on the aromatic region (400 MHz, DMSO-d_6, 25 ºC).



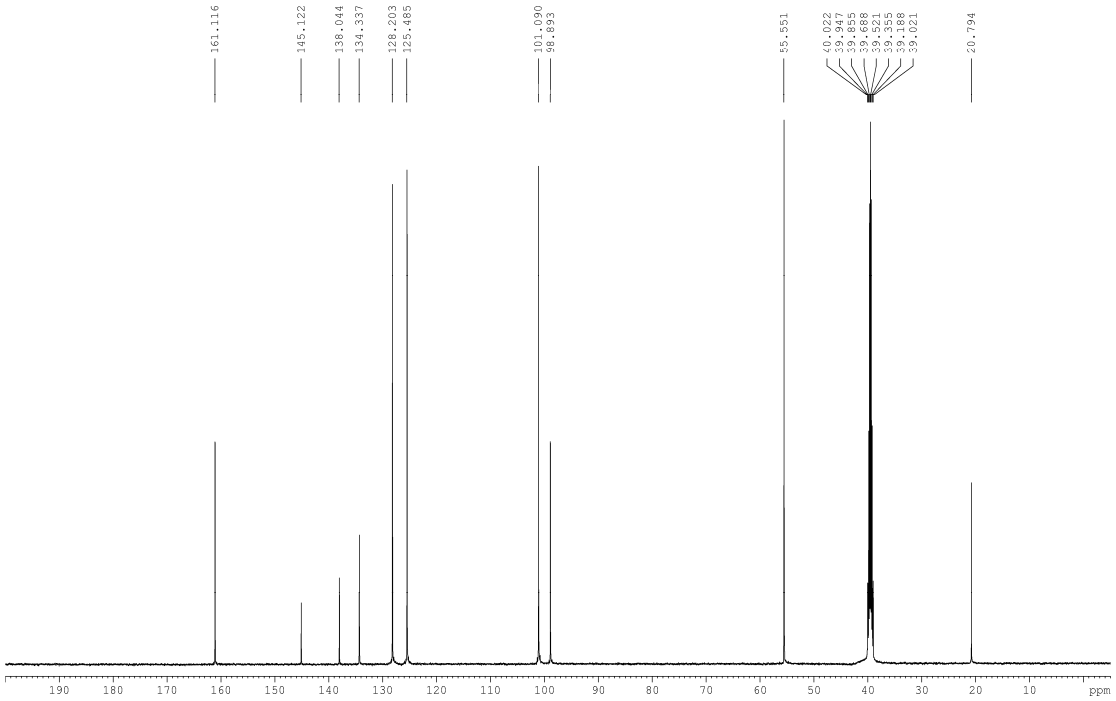
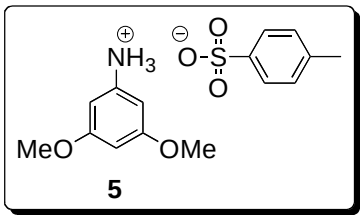


Figure S24. $^{13}C\{^{1}H\}$ NMR spectrum of **5** (125 MHz, DMSO-d_6, 25 ºC).



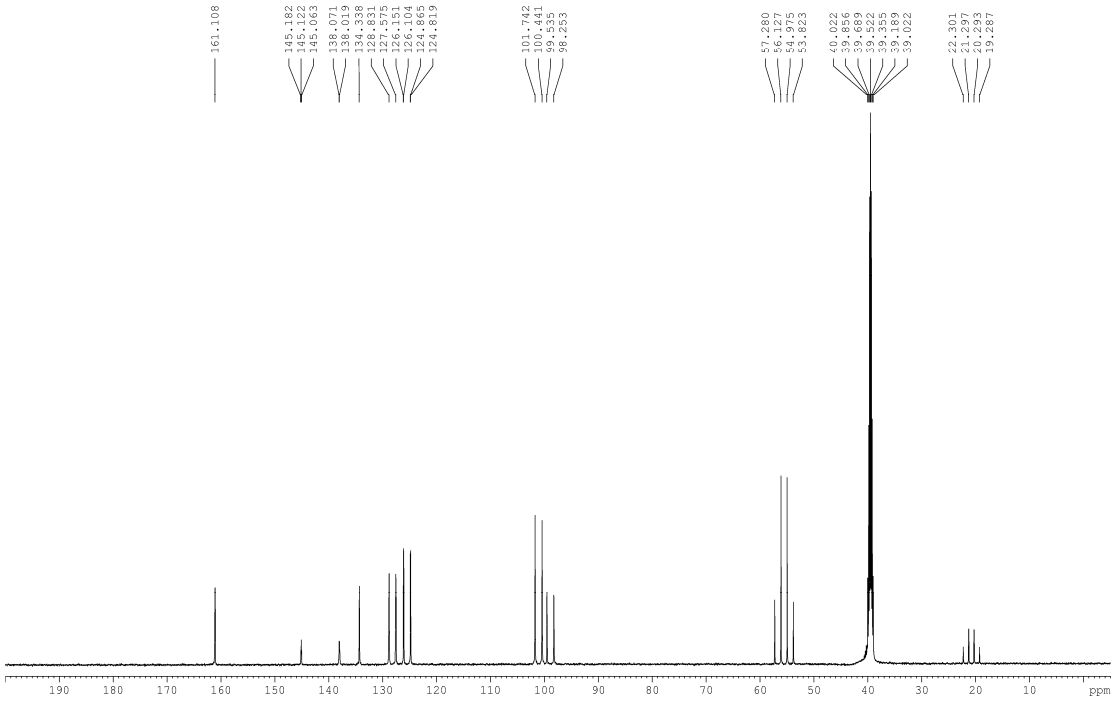
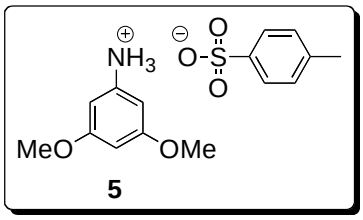


Figure S25. ^{13}C NMR spectrum of **5** (125 MHz, DMSO-d_6, 25 ºC).



5

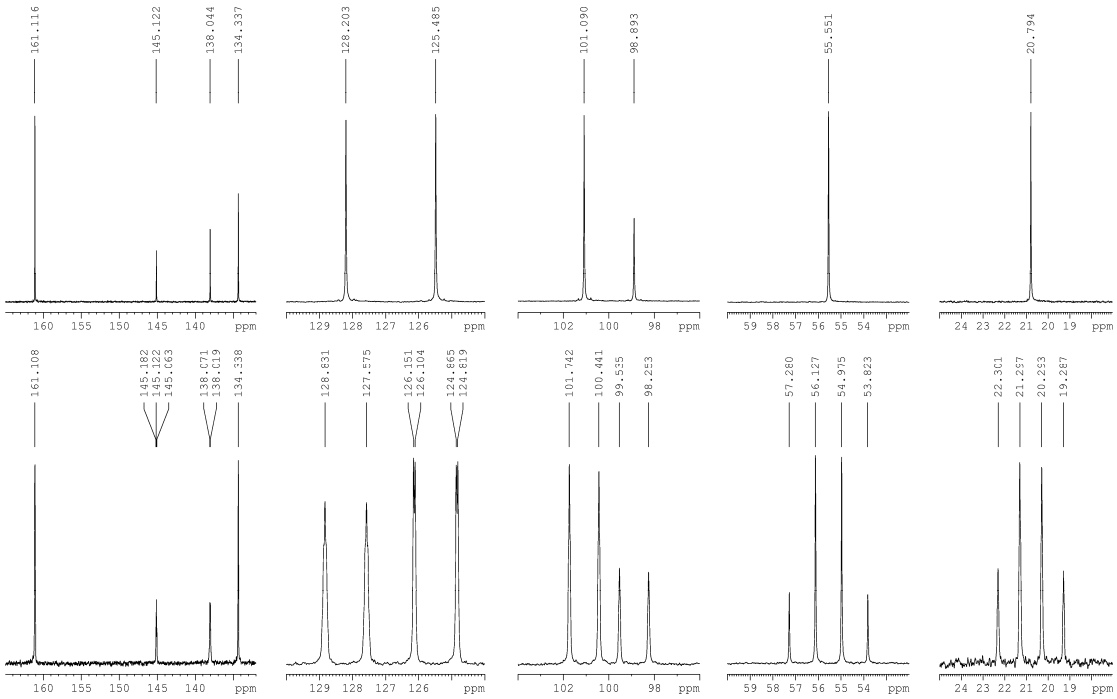


Figure S26. $^{13}C\{^{1}H\}$ and ^{13}C NMR spectra overlay of **5** (125 MHz, DMSO-d_6, 25 ºC). Peak heights are normalized to emphasize splitting.

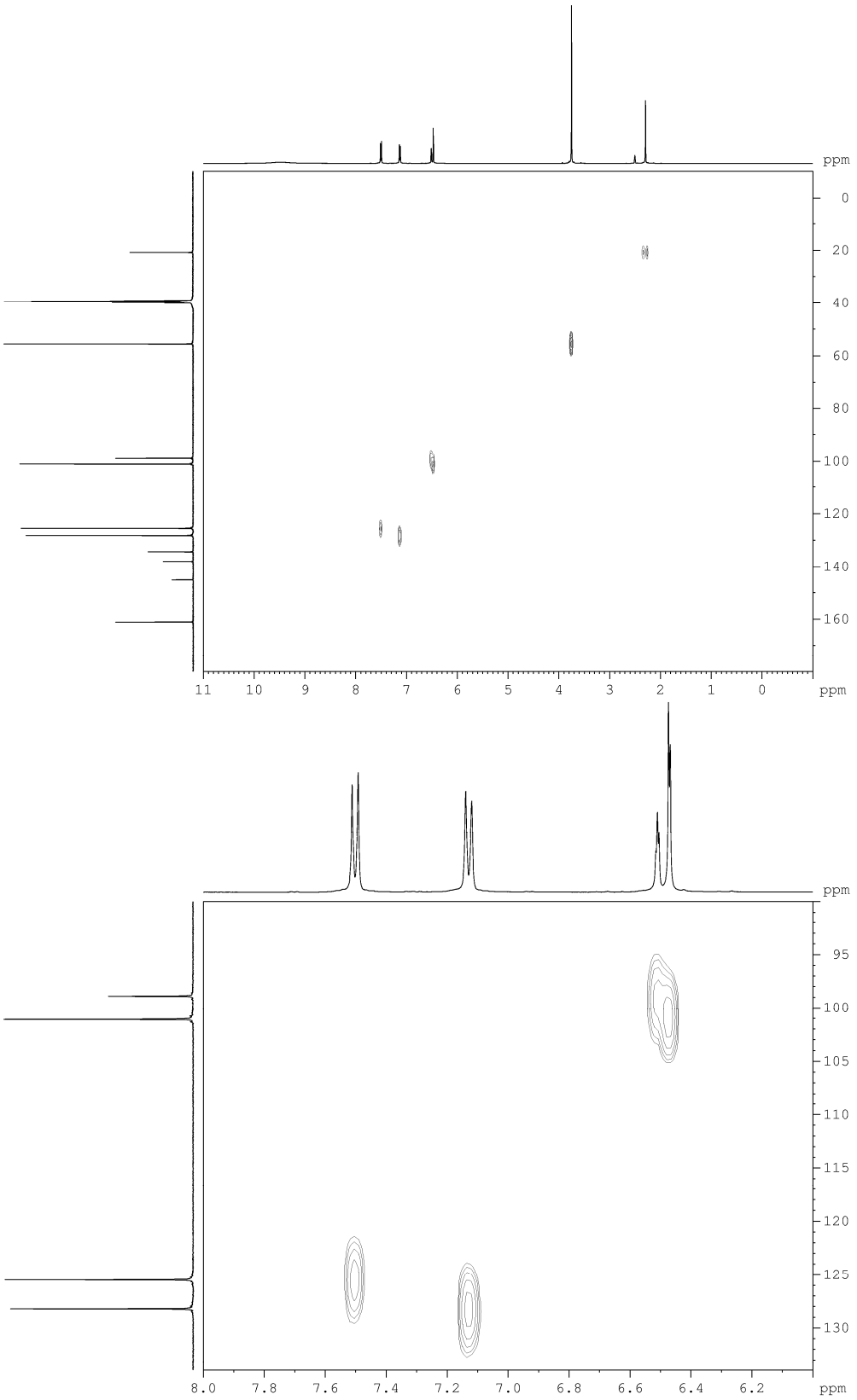


Figure S27. ^{1}H-^{13}C HSQC NMR spectrum of **5** (125 MHz, DMSO-d_6, 25 ºC).

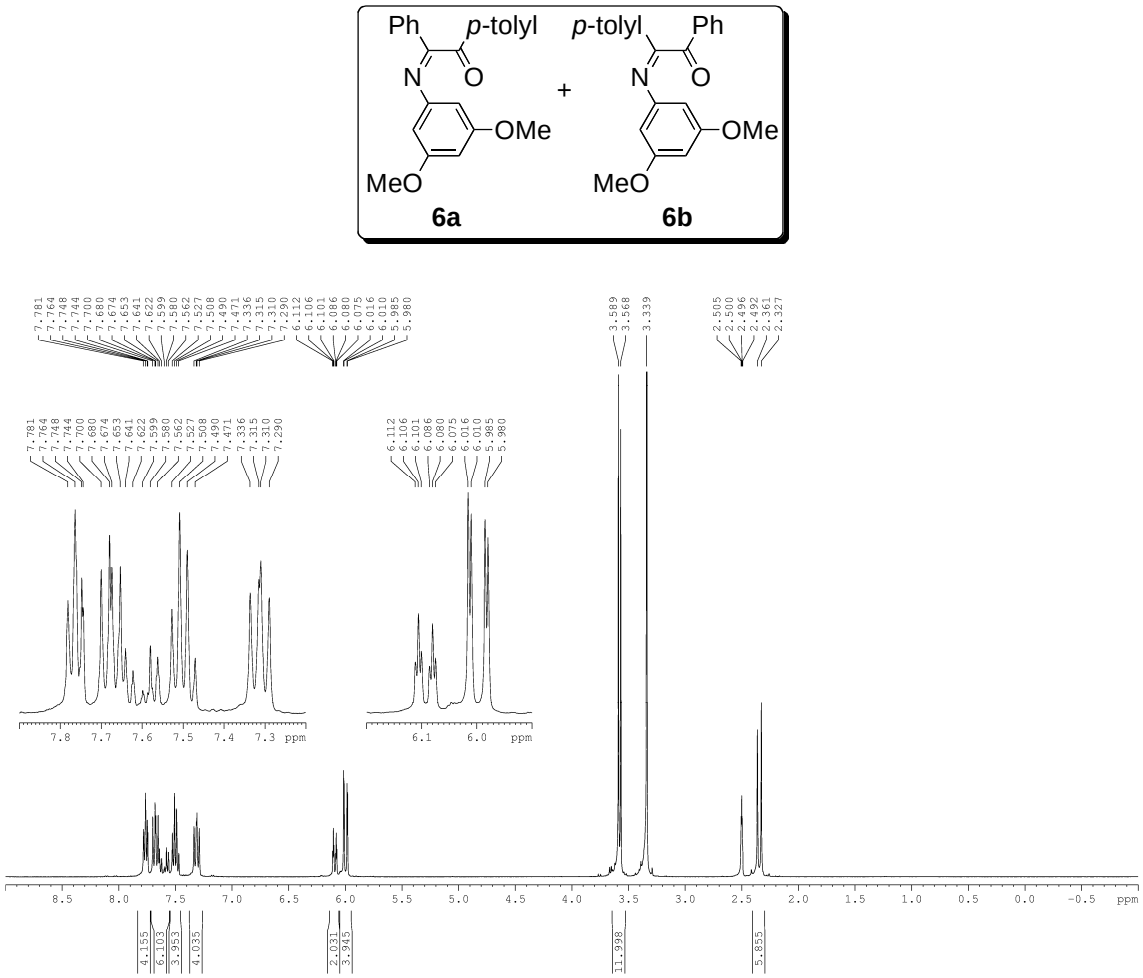
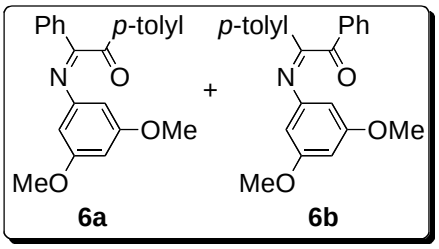


Figure S28. ^{1}H NMR spectrum of **6a** and **6b** (400 MHz, DMSO-d_6, 25 °C).



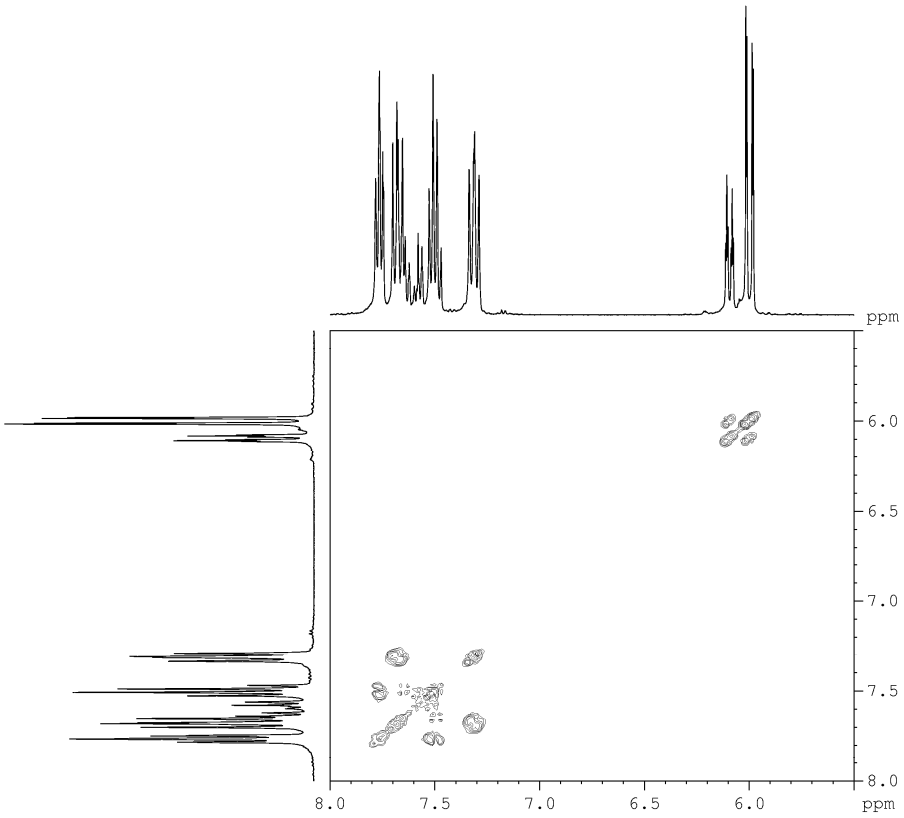
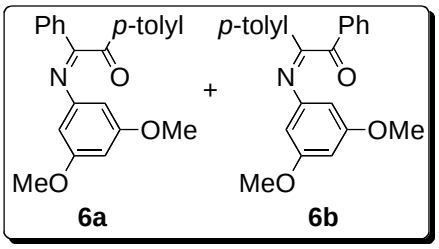


Figure S29. ^{1}H-^{1}H COSY NMR spectrum of **6a** and **6b** zoomed in on the aromatic region (400 MHz, DMSO-d_6, 25 °C).



6a **6b**

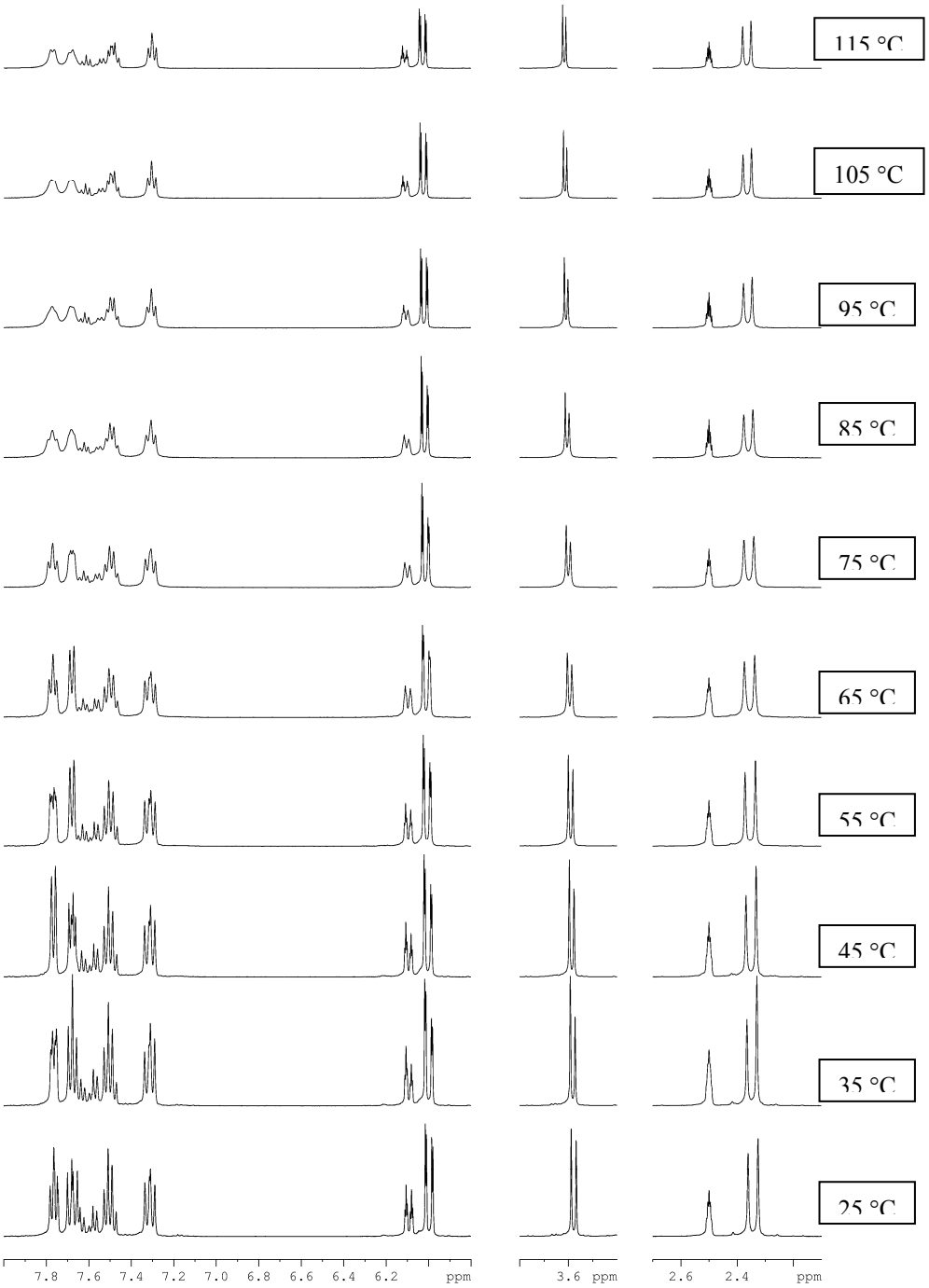


Figure S30. ^{1}H VT NMR spectra of **6a** and **6b** (400 MHz, DMSO-d_6). Peak heights are normalized.

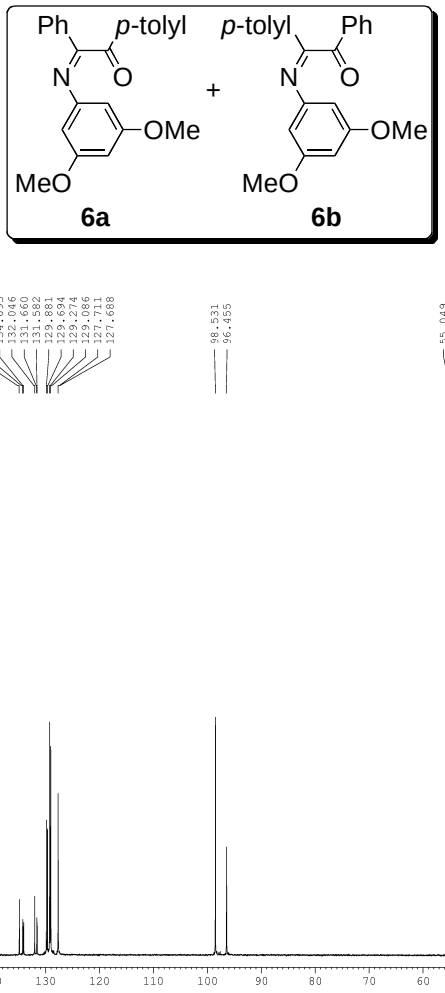
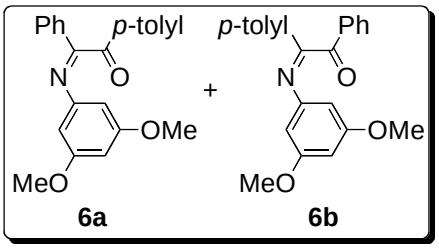


Figure S31. $^{13}C\{^{1}H\}$ NMR spectrum of **6a** and **6b** (125 MHz, DMSO-d_6, 25 ºC).



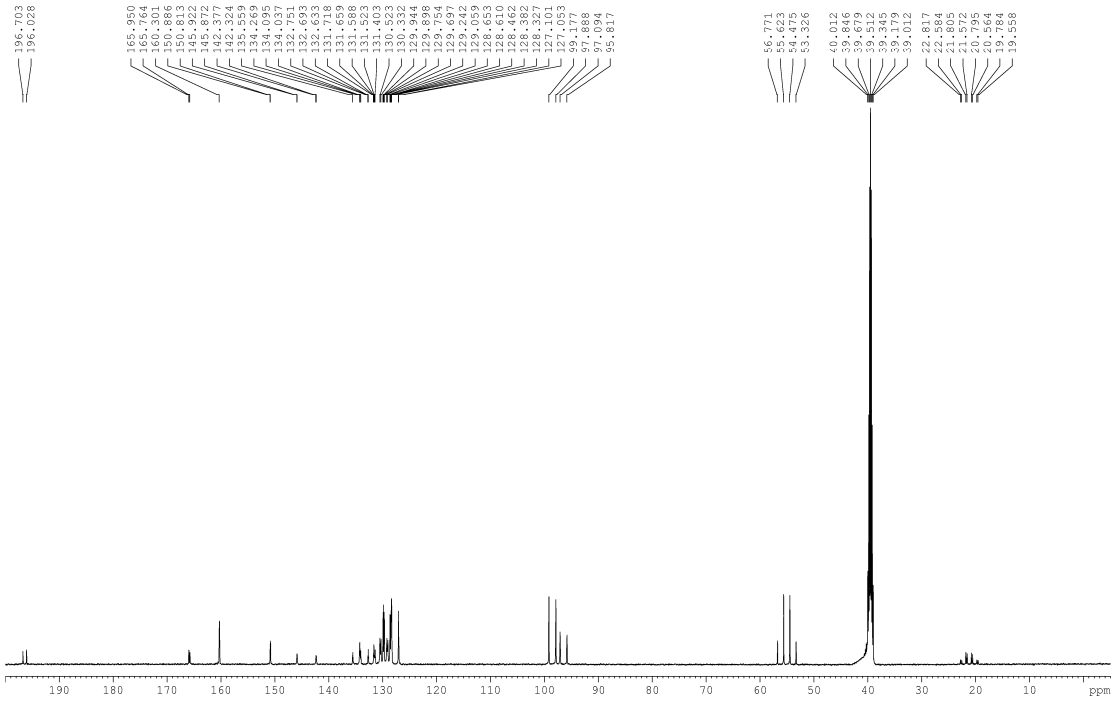
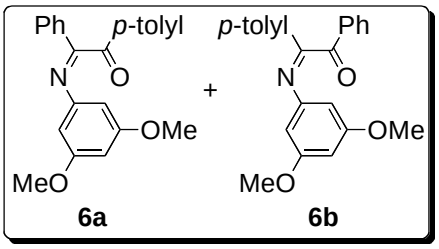


Figure S32. ^{13}C NMR spectrum of **6a** and **6b** (125 MHz, DMSO-d_6, 25 ºC).



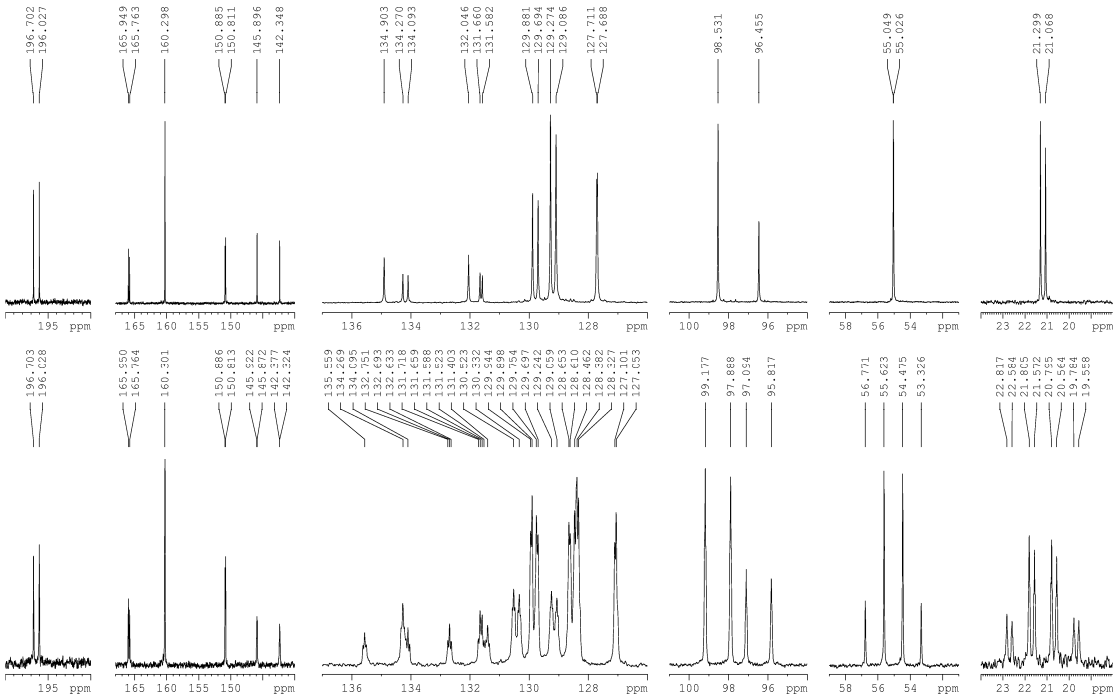


Figure S33. $^{13}C\{^{1}H\}$ and ^{13}C NMR spectra overlay of **6a** and **6b** (125 MHz, DMSO-d_6, 25 ºC). Peak heights are normalized to emphasize splitting.

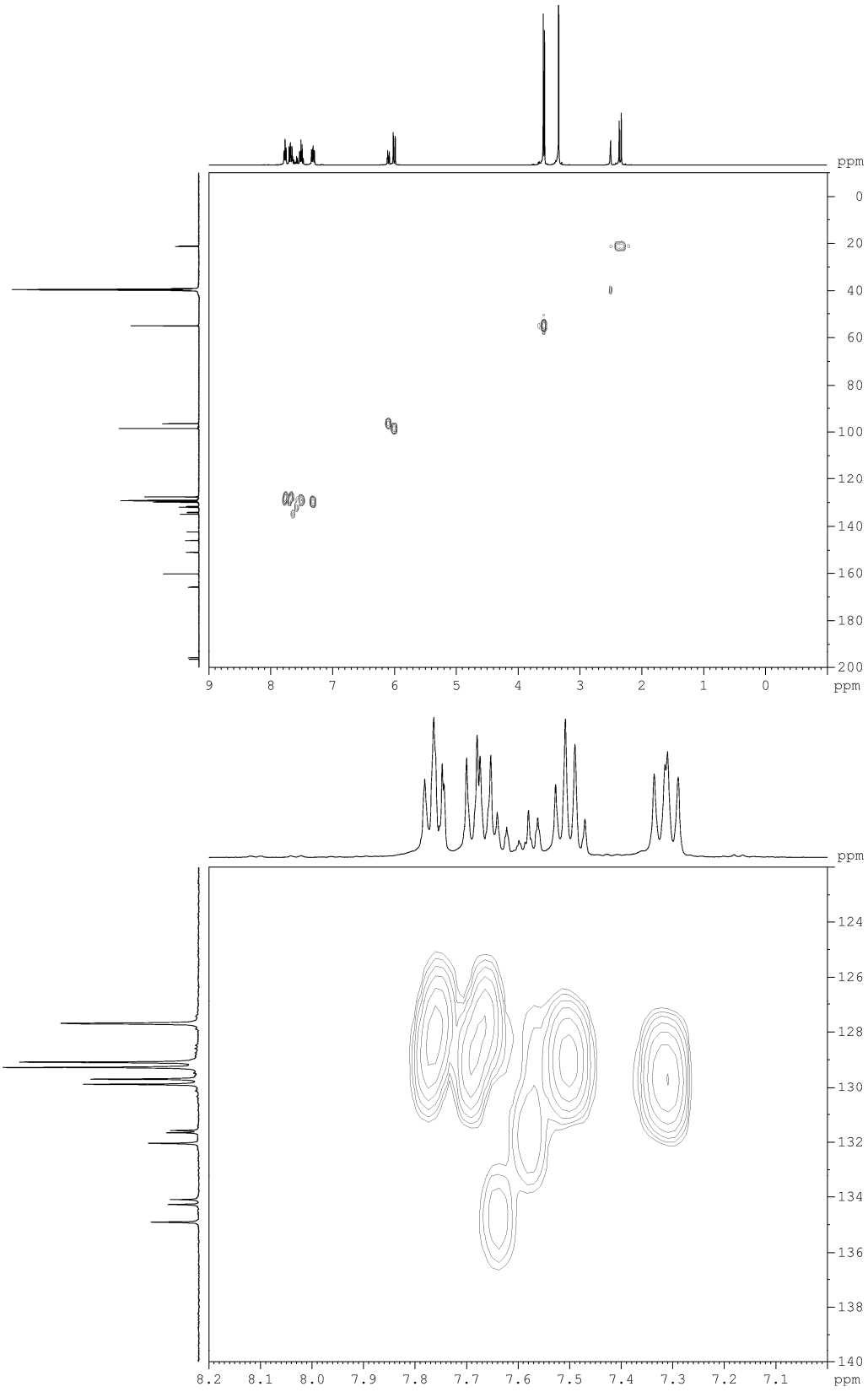


Figure S34. ^{1}H-^{13}C HSQC NMR spectrum of **6a** and **6b** (125 MHz, DMSO-d_6, 25 ºC).

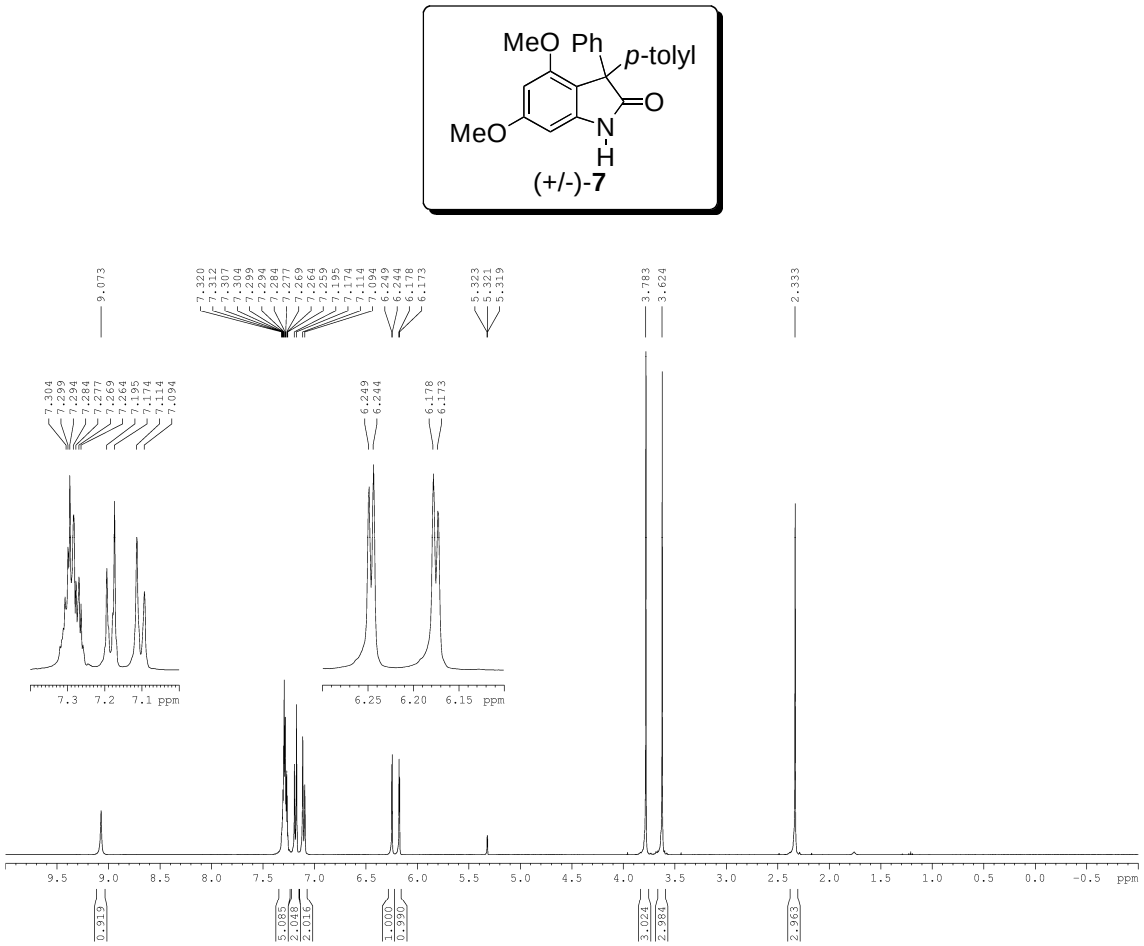


Figure S35. ^{1}H NMR spectrum of (+/-)-**7** (400 MHz, CD_2Cl_2, 25 °C).

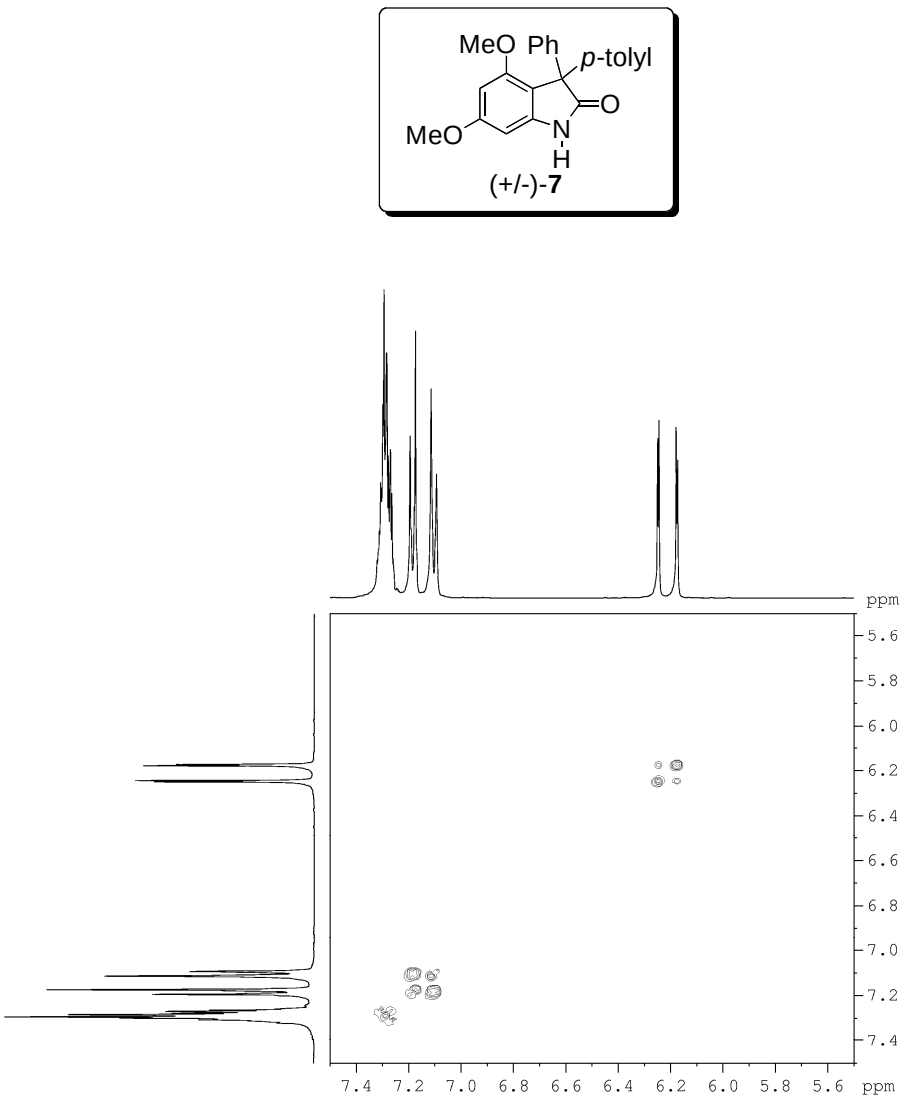


Figure S36. ^{1}H-^{1}H COSY NMR spectrum of (+/-)-**7** zoomed in on the aromatic region (400 MHz, CD_2Cl_2, 25 ºC).

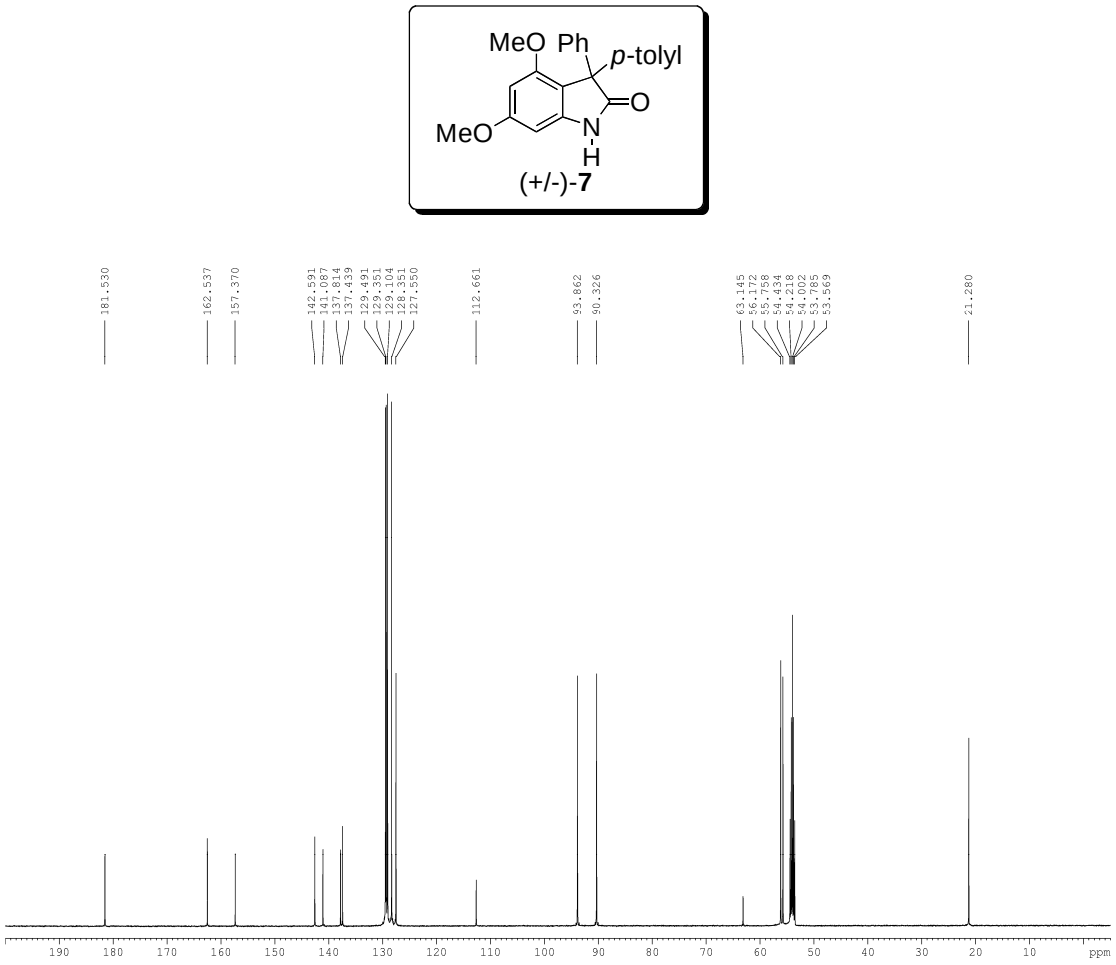


Figure S37. $^{13}C\{^{1}H\}$ NMR spectrum of (+/-)-**7** (125 MHz, CD_2Cl_2, 25 ºC).

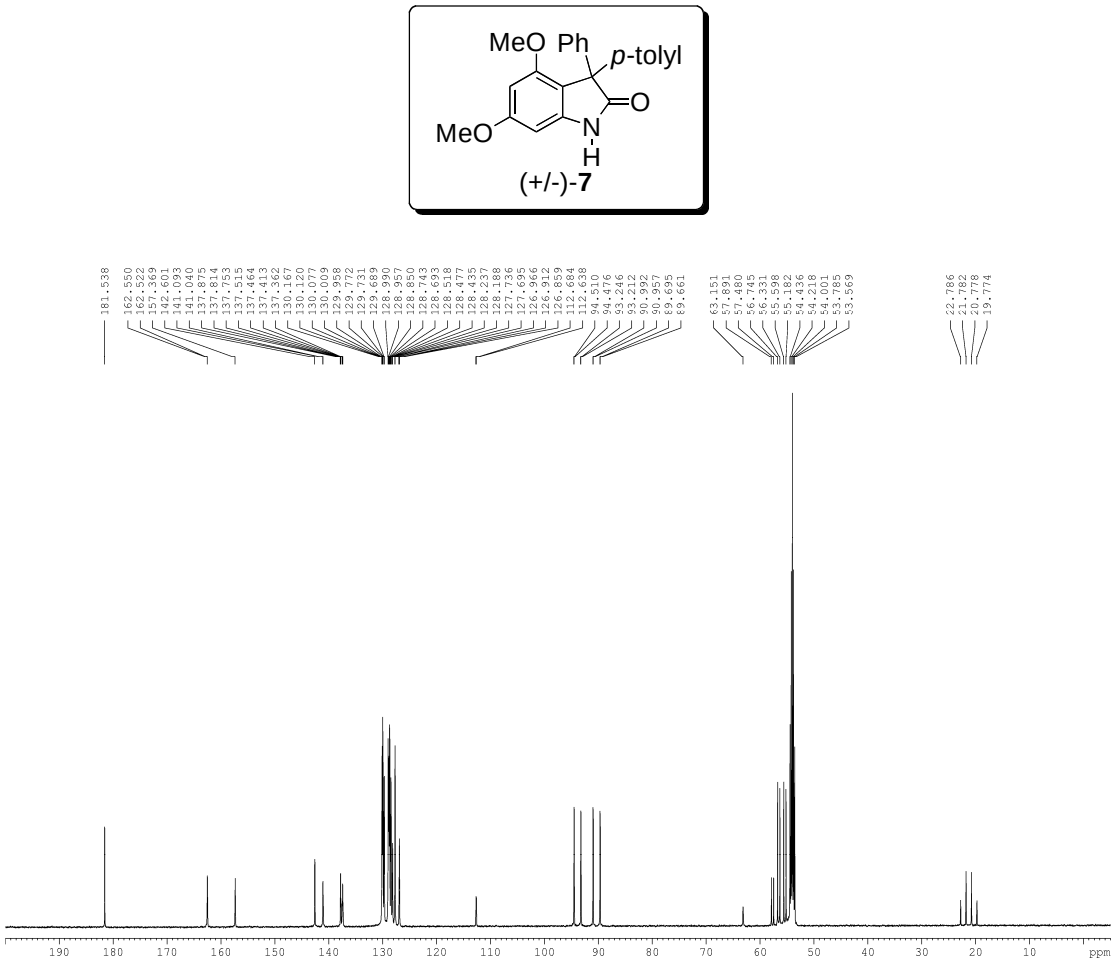


Figure S38. ^{13}C NMR spectrum of (+/-)-**7** (125 MHz, CD_2Cl_2, 25 ºC).

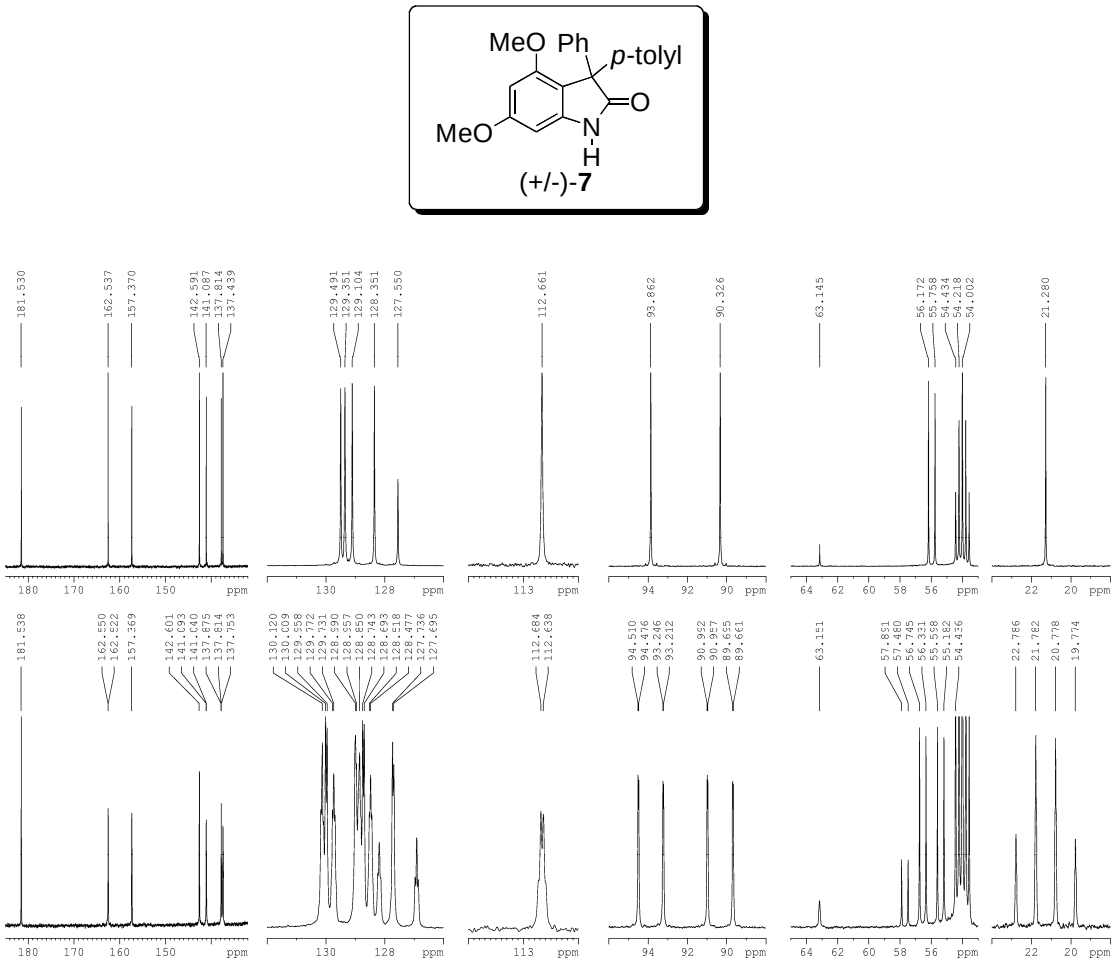


Figure S39. $^{13}C\{^{1}H\}$ and ^{13}C NMR spectra overlay of (+/-)-**7** (125 MHz, CD_2Cl_2, 25 ºC). Peak heights are normalized to emphasize splitting.

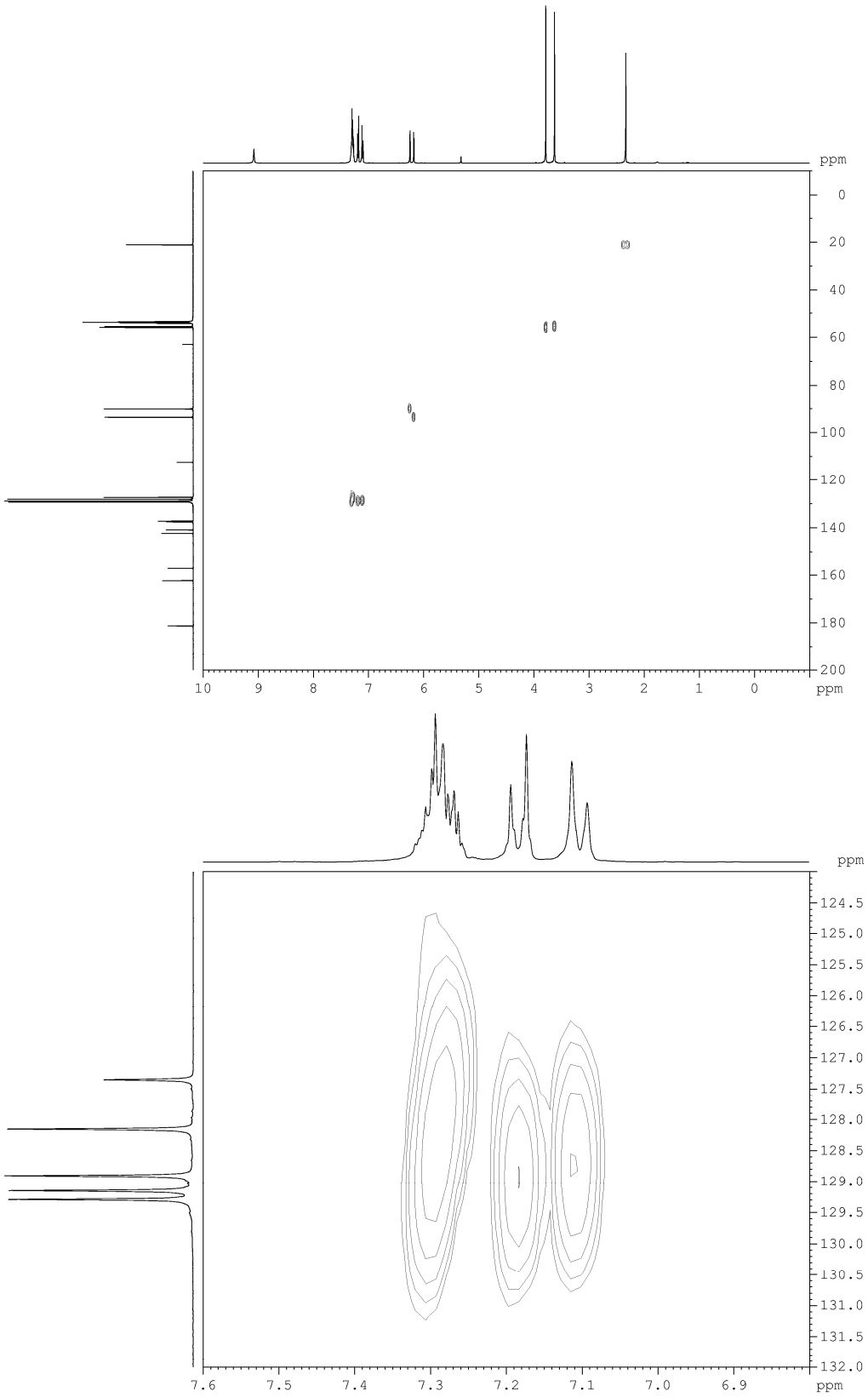


Figure S40. ^{1}H-^{13}C HSQC NMR spectrum of (+/-)-**7** (125 MHz, CD_2Cl_2, 25 ºC).

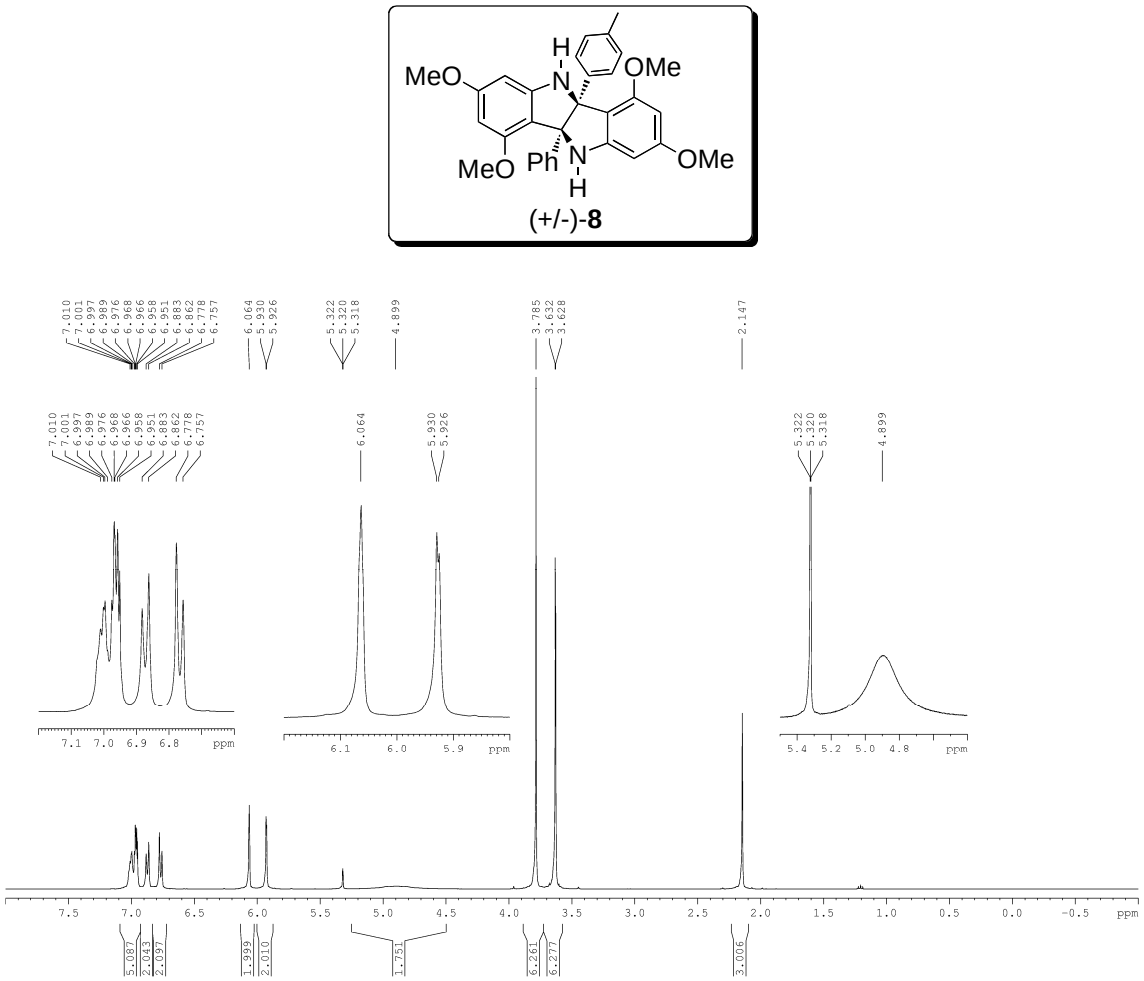
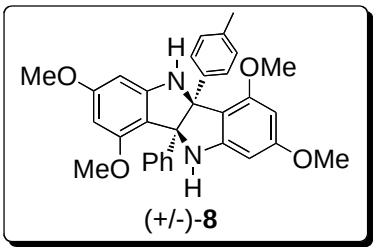


Figure S41. ^{1}H NMR spectrum of (+/-)-**8** (400 MHz, CD_2Cl_2, 25 °C).



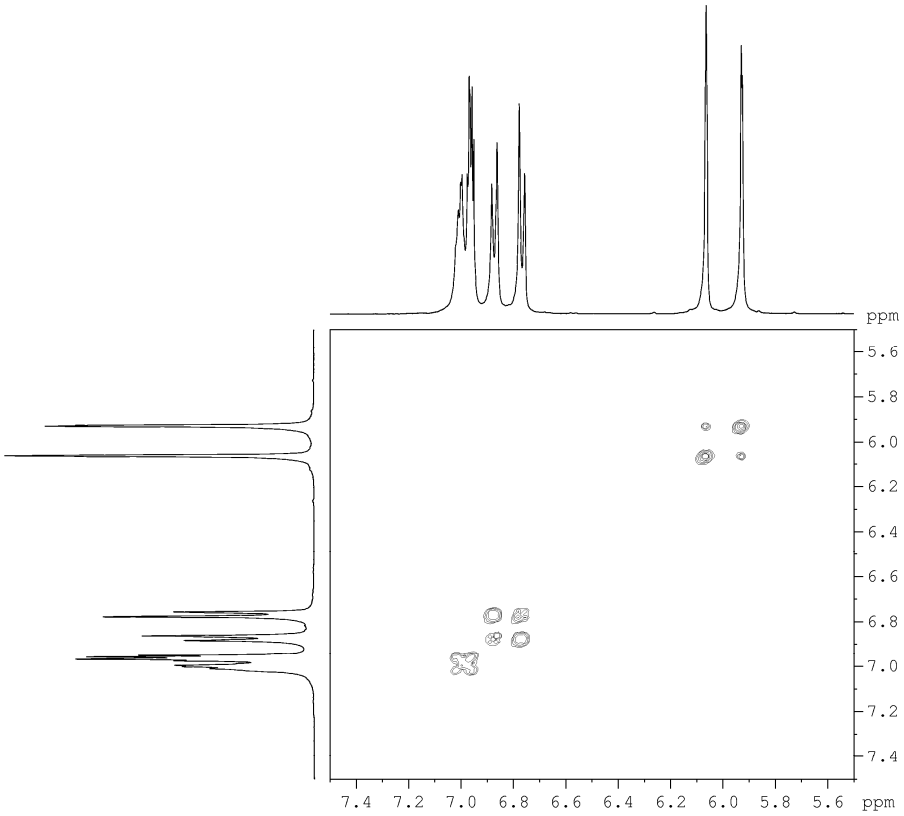


Figure S42. ^{1}H-^{1}H COSY NMR spectrum of (+/-)-**8** zoomed in on the aromatic region (400 MHz, CD_2Cl_2, 25 ºC).

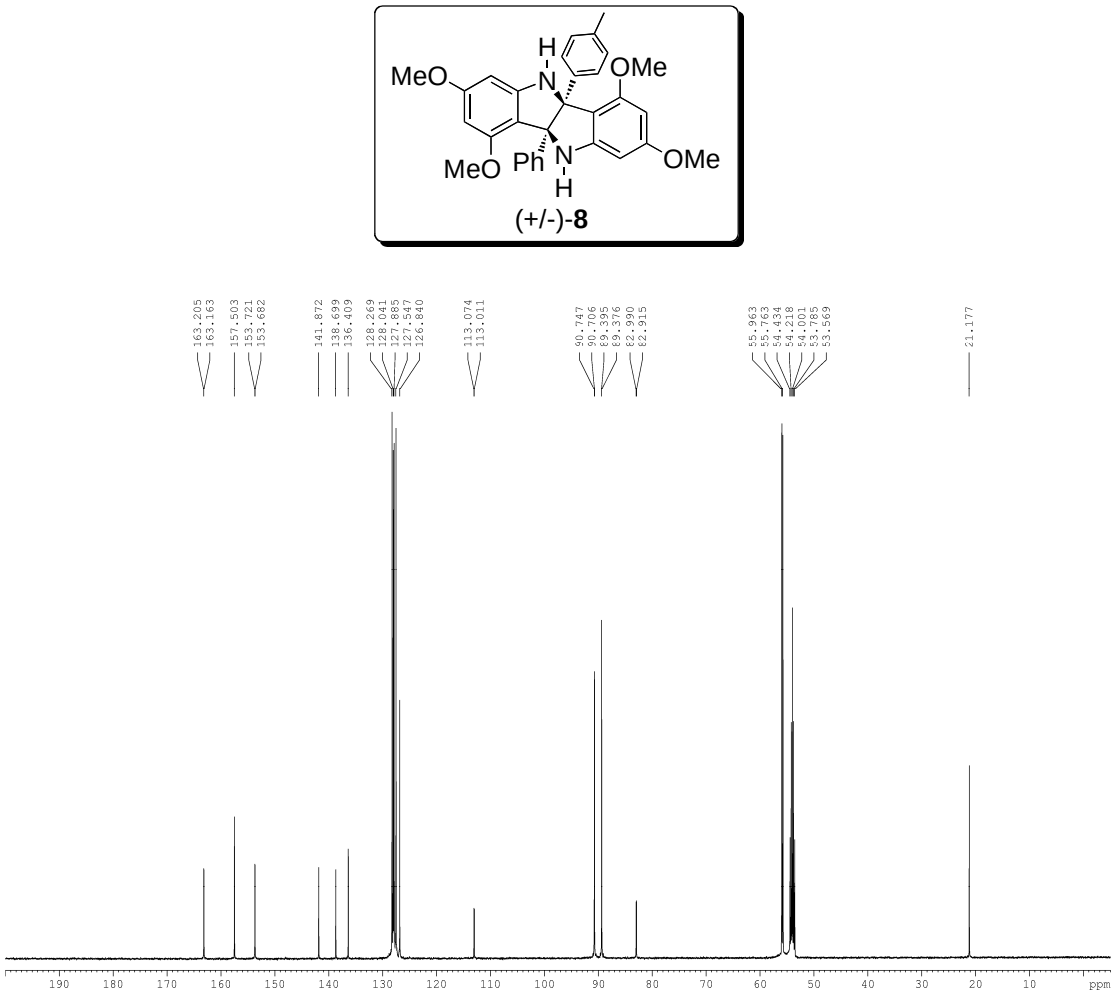


Figure S43. $^{13}C\{^{1}H\}$ NMR spectrum of (+/-)-**8** (125 MHz, CD_2Cl_2, 25 ºC).

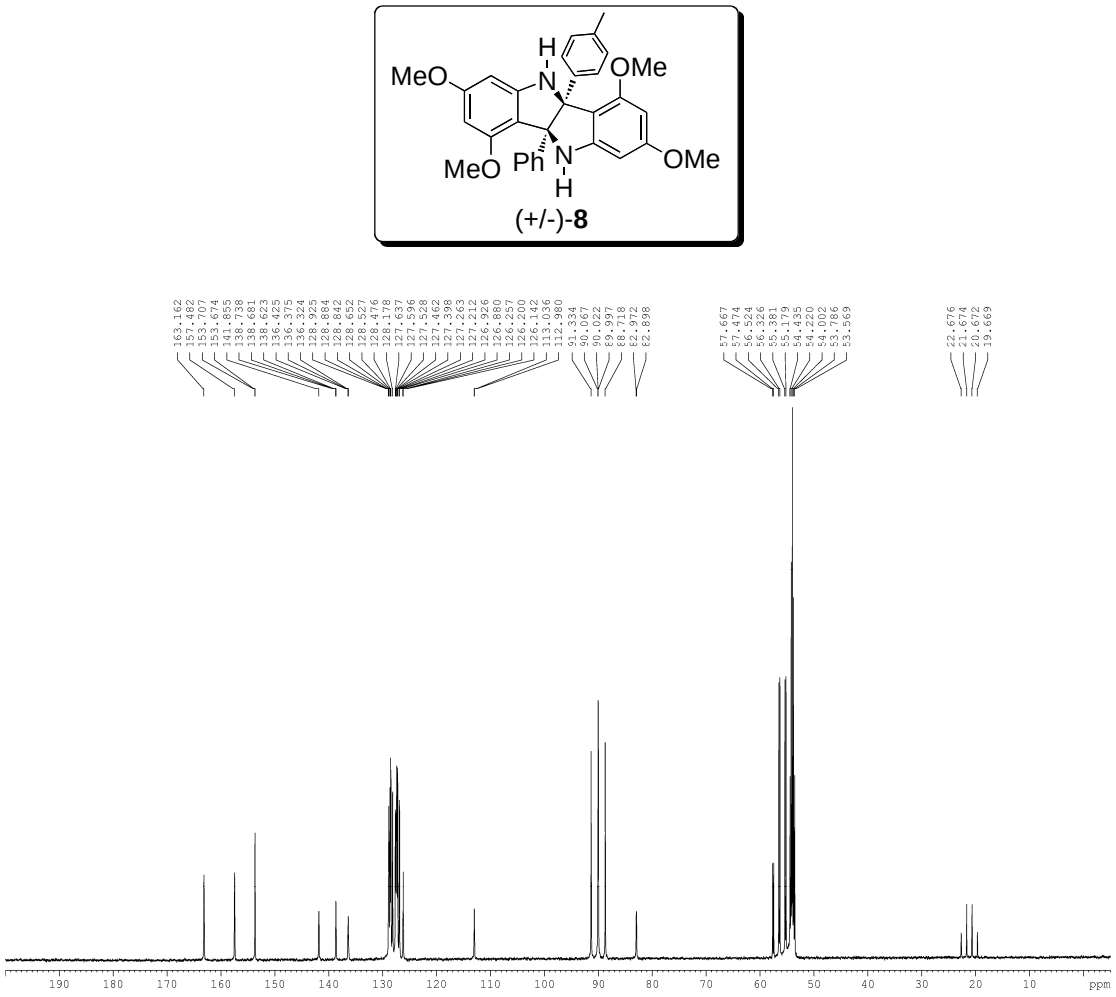
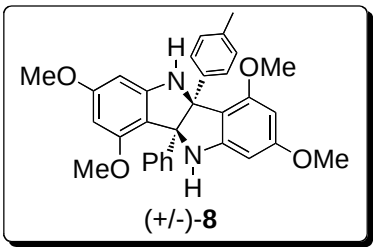


Figure S44. ^{13}C NMR spectrum of (+/-)-**8** (125 MHz, CD_2Cl_2, 25 ºC).



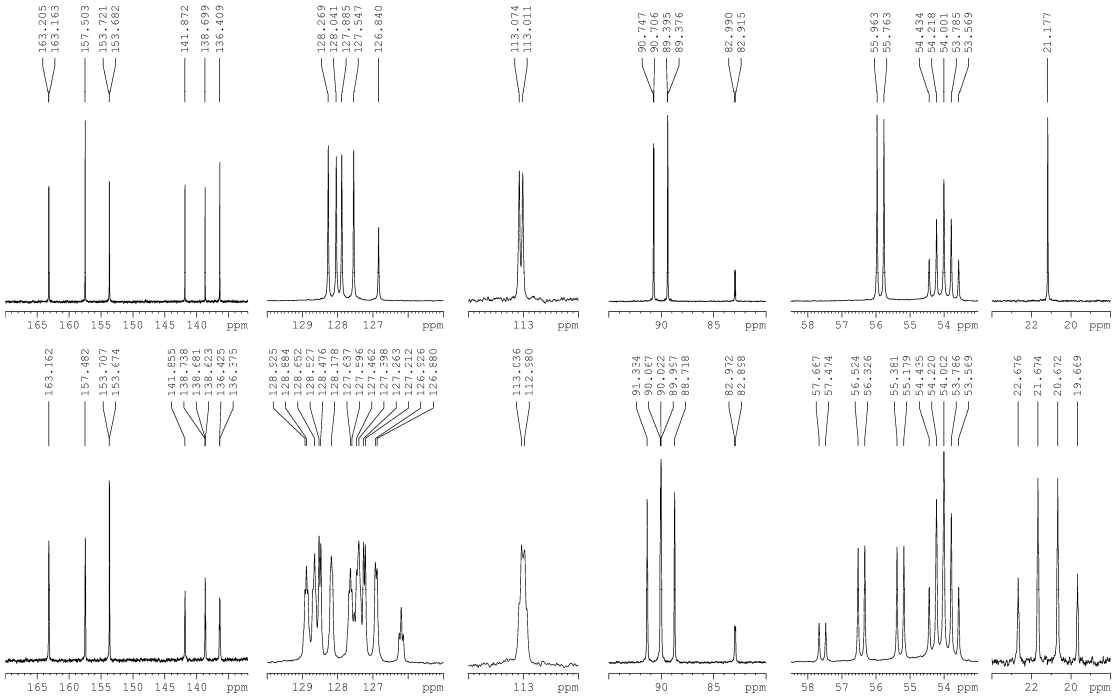


Figure S45. $^{13}C\{^{1}H\}$ and ^{13}C NMR spectra overlay of (+/-)-**8** (125 MHz, CD_2Cl_2, 25 ºC). Peak heights are normalized to emphasize splitting.

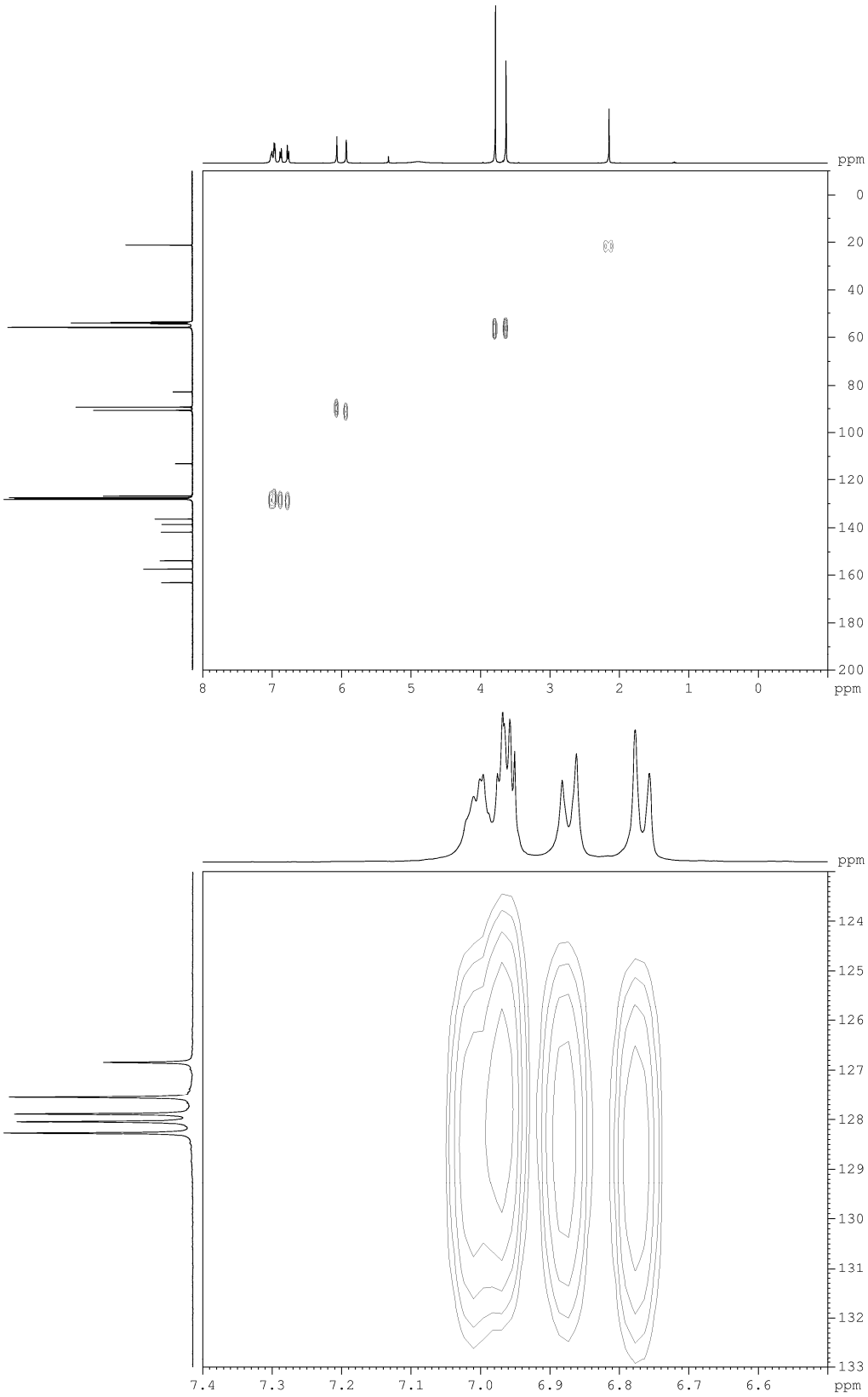


Figure S46. ^{1}H-^{13}C HSQC NMR spectrum of (+/-)-**8** (125 MHz, CD_2Cl_2, 25 ºC).

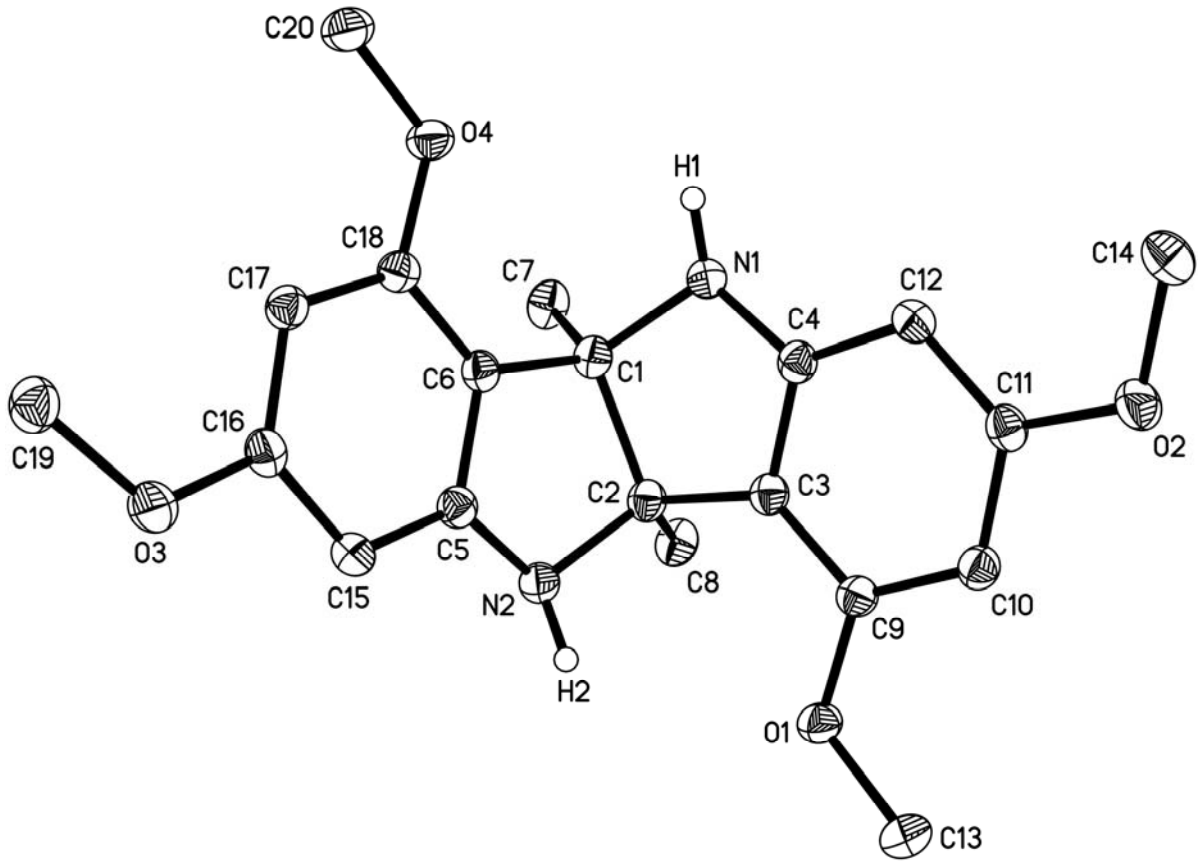


Figure S47. ORTEP diagram of **1** [*Pbca*] with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1 and H2, to emphasize valency of the nitrogen atoms.

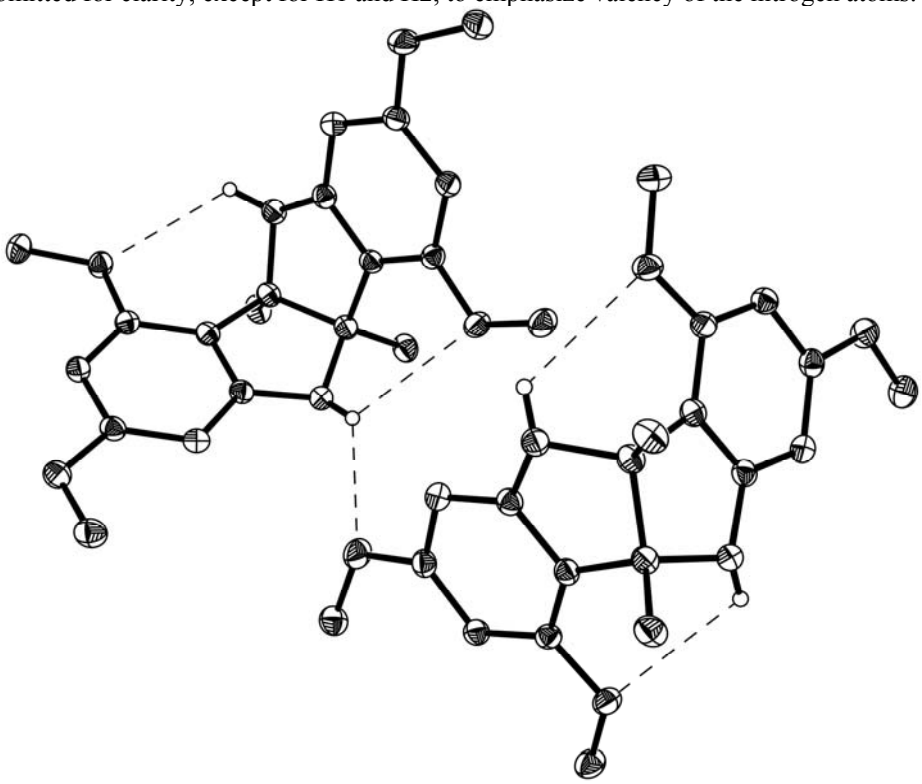


Figure S48. ORTEP diagram of **1** [*Pbca*] with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1, H2, H1A, and H2A, to emphasize intermolecular and intramolecular hydrogen bonding.

Experimental Details for the Structure Determination of (+/-)-**1** [*Pbca*].

Data collection

A crystal (0.38 x 0.22 x 0.16 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 45 seconds and a detector distance of 4.01 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 3990 strong reflections from the actual data collection after integration.[3] See Table S1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group *Pbca* was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were found from the difference Fourier map, and their positional and isotropic displacement parameters were refined independently from those of their respective bonded atoms. The final full matrix least squares refinement converged to $R1 = 0.0527$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1465$ (F^2, all data).

Structure description

The structure is the one suggested, with all atoms in general positions. Weak intermolecular and intramolecular hydrogen bonding is observed (see Figure S48 and Table S7).

References

[1] *APEX2*, version 2011.4-1; Bruker AXS: Madison, WI, 2011.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.68A; Bruker AXS: Madison, WI, 2009.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma|F_o^2|$$

$$R1 = \Sigma||F_o| - |F_c|| \,/\, \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max(0, F_o^2) + 2/3\, F_c^2$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

Table S1. Crystal data and structure refinement for (+/-)-**1** [*Pbca*].

Identification code	(+/-)-**1**, JONJK66	
Empirical formula	C20 H24 N2 O4	
Formula weight	356.41	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	*Pbca*	
Unit cell dimensions	a = 12.226(2) Å	α = 90°
	b = 15.112(3) Å	β = 90°
	c = 18.934(3) Å	γ = 90°
Volume	3498.3(10) Å^3	
Z	8	
Density (calculated)	1.353 Mg/m^3	
Absorption coefficient	0.095 mm^{-1}	
F(000)	1520	
Crystal color, morphology	colorless, needle	
Crystal size	0.38 x 0.22 x 0.16 mm^3	
Theta range for data collection	2.15 to 34.97°	
Index ranges	$-19 \le h \le 19$, $-24 \le k \le 24$, $-30 \le l \le 30$	
Reflections collected	72057	
Independent reflections	7695 [R(int) = 0.0924]	
Observed reflections	4947	
Completeness to theta = 34.97°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9850 and 0.9649	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7695 / 0 / 331	
Goodness-of-fit on F^2	1.036	
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1255	
R indices (all data)	R1 = 0.0914, wR2 = 0.1465	
Largest diff. peak and hole	0.502 and -0.238 e.Å^{-3}	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for (+/-)-**1** [*Pbca*]. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	7277(1)	990(1)	2950(1)	21(1)
O2	9610(1)	-1185(1)	3976(1)	23(1)
O3	7850(1)	4154(1)	5738(1)	25(1)
O4	11342(1)	3374(1)	4678(1)	21(1)
N1	10767(1)	1880(1)	3564(1)	19(1)
N2	8164(1)	2866(1)	3419(1)	19(1)
C1	10129(1)	2695(1)	3429(1)	18(1)
C2	9036(1)	2344(1)	3072(1)	18(1)
C3	9054(1)	1380(1)	3293(1)	17(1)
C4	10055(1)	1166(1)	3592(1)	17(1)
C5	8548(1)	3164(1)	4070(1)	18(1)
C6	9683(1)	3123(1)	4093(1)	17(1)
C7	10806(1)	3329(1)	2979(1)	23(1)
C8	8956(1)	2465(1)	2275(1)	23(1)
C9	8259(1)	726(1)	3228(1)	17(1)
C10	8475(1)	-134(1)	3451(1)	19(1)
C11	9486(1)	-323(1)	3763(1)	18(1)
C12	10291(1)	317(1)	3850(1)	19(1)
C13	6430(1)	341(1)	2924(1)	23(1)
C14	10566(1)	-1387(1)	4376(1)	25(1)
C15	7932(1)	3505(1)	4625(1)	20(1)
C16	8500(1)	3832(1)	5205(1)	19(1)
C17	9639(1)	3823(1)	5242(1)	18(1)
C18	10227(1)	3453(1)	4682(1)	17(1)
C19	8366(1)	4632(1)	6291(1)	24(1)
C20	11907(1)	3679(1)	5291(1)	24(1)

Table S3. Bond lengths [Å] and angles [°] for (+/-)-**1** [*Pbca*].

O(1)-C(9)	1.3704(14)	C(12)-H(12)	0.943(16)
O(1)-C(13)	1.4271(15)	C(13)-H(13A)	0.952(17)
O(2)-C(11)	1.3724(14)	C(13)-H(13B)	1.000(19)
O(2)-C(14)	1.4256(16)	C(13)-H(13C)	0.967(18)
O(3)-C(16)	1.3732(14)	C(14)-H(14A)	0.967(17)
O(3)-C(19)	1.4202(16)	C(14)-H(14B)	0.999(19)
O(4)-C(18)	1.3693(14)	C(14)-H(14C)	1.003(17)
O(4)-C(20)	1.4264(16)	C(15)-C(16)	1.3901(17)
N(1)-C(4)	1.3889(15)	C(15)-H(15)	1.010(16)
N(1)-C(1)	1.4798(16)	C(16)-C(17)	1.3949(17)
N(1)-H(1)	0.878(19)	C(17)-C(18)	1.3984(16)
N(2)-C(5)	1.3952(15)	C(17)-H(17)	0.969(15)
N(2)-C(2)	1.4810(16)	C(19)-H(19A)	0.989(19)
N(2)-H(2)	0.862(17)	C(19)-H(19B)	0.980(18)
C(1)-C(6)	1.5162(16)	C(19)-H(19C)	0.982(17)
C(1)-C(7)	1.5260(17)	C(20)-H(20A)	0.950(17)
C(1)-C(2)	1.5885(16)	C(20)-H(20B)	1.017(17)
C(2)-C(3)	1.5161(17)	C(20)-H(20C)	0.974(18)
C(2)-C(8)	1.5231(17)	C(9)-O(1)-C(13)	116.66(10)
C(3)-C(4)	1.3862(16)	C(11)-O(2)-C(14)	116.78(10)
C(3)-C(9)	1.3917(16)	C(16)-O(3)-C(19)	117.68(10)
C(4)-C(12)	1.4017(17)	C(18)-O(4)-C(20)	116.76(9)
C(5)-C(6)	1.3900(17)	C(4)-N(1)-C(1)	108.82(9)
C(5)-C(15)	1.3916(17)	C(4)-N(1)-H(1)	117.0(11)
C(6)-C(18)	1.3892(16)	C(1)-N(1)-H(1)	118.5(11)
C(7)-H(7A)	0.985(17)	C(5)-N(2)-C(2)	108.74(9)
C(7)-H(7B)	0.999(17)	C(5)-N(2)-H(2)	117.2(11)
C(7)-H(7C)	1.029(17)	C(2)-N(2)-H(2)	112.8(11)
C(8)-H(8A)	0.969(17)	N(1)-C(1)-C(6)	113.62(10)
C(8)-H(8B)	0.976(16)	N(1)-C(1)-C(7)	109.42(10)
C(8)-H(8C)	0.978(17)	C(6)-C(1)-C(7)	113.01(10)
C(9)-C(10)	1.3909(16)	N(1)-C(1)-C(2)	103.84(9)
C(10)-C(11)	1.3997(17)	C(6)-C(1)-C(2)	101.15(9)
C(10)-H(10)	0.977(16)	C(7)-C(1)-C(2)	115.36(10)
C(11)-C(12)	1.3900(17)	N(2)-C(2)-C(3)	113.59(10)

N(2)-C(2)-C(8)	109.18(10)	C(12)-C(11)-C(10)	122.26(11)
C(3)-C(2)-C(8)	112.99(10)	C(11)-C(12)-C(4)	116.69(11)
N(2)-C(2)-C(1)	103.83(9)	C(11)-C(12)-H(12)	123.1(10)
C(3)-C(2)-C(1)	101.04(9)	C(4)-C(12)-H(12)	120.2(10)
C(8)-C(2)-C(1)	115.80(10)	O(1)-C(13)-H(13A)	104.4(10)
C(4)-C(3)-C(9)	119.14(11)	O(1)-C(13)-H(13B)	113.1(10)
C(4)-C(3)-C(2)	110.47(10)	H(13A)-C(13)-H(13B)	112.5(14)
C(9)-C(3)-C(2)	130.38(10)	O(1)-C(13)-H(13C)	111.3(10)
C(3)-C(4)-N(1)	110.88(10)	H(13A)-C(13)-H(13C)	111.5(15)
C(3)-C(4)-C(12)	122.51(11)	H(13B)-C(13)-H(13C)	104.2(14)
N(1)-C(4)-C(12)	126.53(11)	O(2)-C(14)-H(14A)	106.4(10)
C(6)-C(5)-C(15)	122.24(11)	O(2)-C(14)-H(14B)	111.1(11)
C(6)-C(5)-N(2)	110.48(10)	H(14A)-C(14)-H(14B)	108.1(15)
C(15)-C(5)-N(2)	127.20(11)	O(2)-C(14)-H(14C)	111.0(10)
C(18)-C(6)-C(5)	119.12(11)	H(14A)-C(14)-H(14C)	108.2(14)
C(18)-C(6)-C(1)	130.28(11)	H(14B)-C(14)-H(14C)	111.9(15)
C(5)-C(6)-C(1)	110.60(10)	C(16)-C(15)-C(5)	117.20(11)
C(1)-C(7)-H(7A)	108.7(9)	C(16)-C(15)-H(15)	121.8(9)
C(1)-C(7)-H(7B)	110.7(10)	C(5)-C(15)-H(15)	121.0(10)
H(7A)-C(7)-H(7B)	111.4(14)	O(3)-C(16)-C(15)	114.63(11)
C(1)-C(7)-H(7C)	110.0(9)	O(3)-C(16)-C(17)	122.98(11)
H(7A)-C(7)-H(7C)	107.3(13)	C(15)-C(16)-C(17)	122.38(11)
H(7B)-C(7)-H(7C)	108.7(13)	C(16)-C(17)-C(18)	118.58(11)
C(2)-C(8)-H(8A)	111.5(10)	C(16)-C(17)-H(17)	122.2(9)
C(2)-C(8)-H(8B)	110.5(9)	C(18)-C(17)-H(17)	119.2(9)
H(8A)-C(8)-H(8B)	109.4(14)	O(4)-C(18)-C(6)	116.21(10)
C(2)-C(8)-H(8C)	113.2(10)	O(4)-C(18)-C(17)	123.35(10)
H(8A)-C(8)-H(8C)	105.9(14)	C(6)-C(18)-C(17)	120.43(11)
H(8B)-C(8)-H(8C)	106.2(13)	O(3)-C(19)-H(19A)	103.9(11)
O(1)-C(9)-C(10)	123.71(10)	O(3)-C(19)-H(19B)	109.6(10)
O(1)-C(9)-C(3)	116.02(10)	H(19A)-C(19)-H(19B)	109.5(15)
C(10)-C(9)-C(3)	120.26(11)	O(3)-C(19)-H(19C)	110.5(9)
C(9)-C(10)-C(11)	119.07(11)	H(19A)-C(19)-H(19C)	112.1(15)
C(9)-C(10)-H(10)	122.1(9)	H(19B)-C(19)-H(19C)	110.9(14)
C(11)-C(10)-H(10)	118.8(9)	O(4)-C(20)-H(20A)	106.4(10)
O(2)-C(11)-C(12)	123.18(11)	O(4)-C(20)-H(20B)	111.7(9)
O(2)-C(11)-C(10)	114.55(10)	H(20A)-C(20)-H(20B)	107.8(13)

O(4)-C(20)-H(20C)	110.9(10)	H(20B)-C(20)-H(20C)	109.9(13)
H(20A)-C(20)-H(20C)	110.1(14)		

Table S4. Anisotropic displacement parameters ($Å^2$ x 10^3) for (+/-)-**1** [*Pbca*]. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$h^2 a^{*2} U_{11}$ + ... + 2 h k a* b* U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	18(1)	20(1)	23(1)	1(1)	-4(1)	-1(1)
O2	22(1)	17(1)	30(1)	5(1)	0(1)	2(1)
O3	21(1)	32(1)	23(1)	-8(1)	4(1)	2(1)
O4	16(1)	26(1)	20(1)	-3(1)	0(1)	-1(1)
N1	16(1)	17(1)	24(1)	-2(1)	0(1)	0(1)
N2	19(1)	18(1)	20(1)	-1(1)	-3(1)	1(1)
C1	20(1)	17(1)	18(1)	0(1)	1(1)	-1(1)
C2	21(1)	17(1)	17(1)	0(1)	0(1)	0(1)
C3	19(1)	17(1)	15(1)	-1(1)	0(1)	0(1)
C4	18(1)	17(1)	16(1)	-2(1)	2(1)	0(1)
C5	19(1)	15(1)	18(1)	1(1)	-1(1)	0(1)
C6	18(1)	14(1)	18(1)	0(1)	1(1)	0(1)
C7	27(1)	21(1)	21(1)	0(1)	6(1)	-4(1)
C8	30(1)	22(1)	17(1)	1(1)	-2(1)	-2(1)
C9	18(1)	19(1)	15(1)	-2(1)	-1(1)	0(1)
C10	19(1)	17(1)	20(1)	0(1)	1(1)	-1(1)
C11	20(1)	16(1)	19(1)	2(1)	3(1)	2(1)
C12	18(1)	19(1)	19(1)	0(1)	1(1)	2(1)
C13	20(1)	26(1)	23(1)	-1(1)	-3(1)	-4(1)
C14	26(1)	23(1)	26(1)	4(1)	-1(1)	5(1)
C15	18(1)	19(1)	22(1)	-1(1)	0(1)	2(1)
C16	20(1)	17(1)	20(1)	-1(1)	3(1)	1(1)
C17	19(1)	18(1)	18(1)	-2(1)	1(1)	0(1)
C18	17(1)	16(1)	18(1)	2(1)	0(1)	-1(1)
C19	26(1)	25(1)	20(1)	-4(1)	2(1)	0(1)
C20	19(1)	31(1)	23(1)	-4(1)	-3(1)	0(1)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for (+/-)-**1** [*Pbca*].

	x	y	z	$U_{(eq)}$
H1	11290(15)	1898(11)	3881(9)	31(4)
H2	7539(14)	2603(11)	3414(9)	27(4)
H7A	11485(14)	3472(11)	3234(9)	26(4)
H7B	10379(13)	3874(12)	2866(9)	29(4)
H7C	11027(13)	3027(11)	2513(9)	27(4)
H8A	8275(14)	2231(11)	2091(9)	30(4)
H8B	9569(13)	2175(11)	2039(8)	22(4)
H8C	8979(13)	3086(11)	2129(9)	25(4)
H10	7934(13)	-607(10)	3410(8)	23(4)
H12	10966(13)	204(10)	4074(9)	21(4)
H13A	5822(13)	645(12)	2724(10)	30(4)
H13B	6260(14)	79(12)	3396(10)	33(5)
H13C	6643(14)	-160(12)	2637(9)	33(4)
H14A	10517(14)	-2007(11)	4496(9)	27(4)
H14B	10586(15)	-1040(12)	4825(10)	37(5)
H14C	11244(14)	-1296(11)	4087(9)	27(4)
H15	7107(13)	3509(10)	4605(9)	24(4)
H17	10033(13)	4060(10)	5644(8)	21(4)
H19A	7749(15)	4873(12)	6569(10)	37(5)
H19B	8792(14)	5121(12)	6091(9)	30(4)
H19C	8833(13)	4238(11)	6572(9)	24(4)
H20A	12649(14)	3505(11)	5236(9)	26(4)
H20B	11617(13)	3389(10)	5738(9)	24(4)
H20C	11857(14)	4320(12)	5332(9)	30(4)

Table S6. Torsion angles [°] for (+/-)-**1** [*Pbca*].

C4-N1-C1-C6	86.33(12)	C2-C1-C6-C18	170.44(12)
C4-N1-C1-C7	-146.32(10)	N1-C1-C6-C5	-119.97(11)
C4-N1-C1-C2	-22.64(12)	C7-C1-C6-C5	114.58(11)
C5-N2-C2-C3	85.49(12)	C2-C1-C6-C5	-9.34(12)
C5-N2-C2-C8	-147.41(10)	C13-O1-C9-C10	2.66(16)
C5-N2-C2-C1	-23.33(12)	C13-O1-C9-C3	-175.97(10)
N1-C1-C2-N2	137.03(9)	C4-C3-C9-O1	177.78(10)
C6-C1-C2-N2	19.04(11)	C2-C3-C9-O1	-3.53(18)
C7-C1-C2-N2	-103.26(11)	C4-C3-C9-C10	-0.91(17)
N1-C1-C2-C3	19.12(11)	C2-C3-C9-C10	177.79(11)
C6-C1-C2-C3	-98.86(10)	O1-C9-C10-C11	-176.46(11)
C7-C1-C2-C3	138.84(10)	C3-C9-C10-C11	2.12(17)
N1-C1-C2-C8	-103.31(11)	C14-O2-C11-C12	7.41(17)
C6-C1-C2-C8	138.70(10)	C14-O2-C11-C10	-172.33(11)
C7-C1-C2-C8	16.40(15)	C9-C10-C11-O2	178.88(10)
N2-C2-C3-C4	-120.76(11)	C9-C10-C11-C12	-0.87(18)
C8-C2-C3-C4	114.16(12)	O2-C11-C12-C4	178.74(11)
C1-C2-C3-C4	-10.21(12)	C10-C11-C12-C4	-1.53(17)
N2-C2-C3-C9	60.46(16)	C3-C4-C12-C11	2.82(17)
C8-C2-C3-C9	-64.62(16)	N1-C4-C12-C11	-173.79(11)
C1-C2-C3-C9	171.01(12)	C6-C5-C15-C16	1.74(17)
C9-C3-C4-N1	175.44(10)	N2-C5-C15-C16	-174.67(11)
C2-C3-C4-N1	-3.50(13)	C19-O3-C16-C15	-169.52(11)
C9-C3-C4-C12	-1.65(17)	C19-O3-C16-C17	11.09(17)
C2-C3-C4-C12	179.41(10)	C5-C15-C16-O3	-179.53(10)
C1-N1-C4-C3	17.14(13)	C5-C15-C16-C17	-0.14(18)
C1-N1-C4-C12	-165.92(11)	O3-C16-C17-C18	177.83(11)
C2-N2-C5-C6	18.41(13)	C15-C16-C17-C18	-1.51(18)
C2-N2-C5-C15	-164.83(11)	C20-O4-C18-C6	-177.66(11)
C15-C5-C6-C18	-1.63(17)	C20-O4-C18-C17	0.96(16)
N2-C5-C6-C18	175.32(10)	C5-C6-C18-O4	178.54(10)
C15-C5-C6-C1	178.18(10)	C1-C6-C18-O4	-1.23(17)
N2-C5-C6-C1	-4.87(13)	C5-C6-C18-C17	-0.12(17)
N1-C1-C6-C18	59.82(16)	C1-C6-C18-C17	-179.88(11)
C7-C1-C6-C18	-65.63(16)	C16-C17-C18-O4	-176.93(11)

C16-C17-C18-C6	1.63(17)

Table S7. Hydrogen bonds and close contacts for (+/-)-**1** [*Pbca*] [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1...O4	0.878(19)	2.694(17)	3.1685(14)	115.2(14)
N1-H1...O3#1	0.878(19)	2.585(18)	3.2670(15)	135.2(14)
N2-H2...O1	0.862(17)	2.609(17)	3.1618(14)	122.9(14)

Symmetry transformations used to generate equivalent atoms:

#1 x+1/2,-y+1/2,-z+1

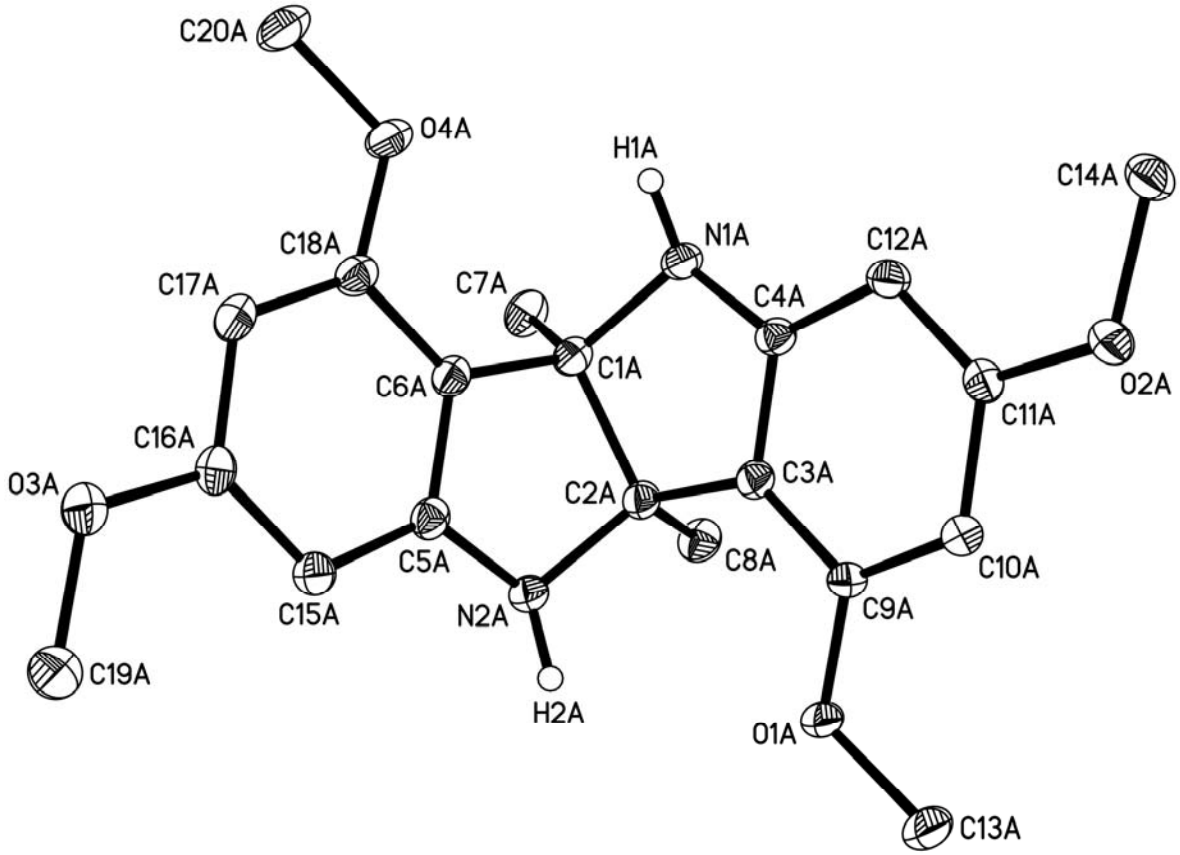


Figure S49. ORTEP diagram of **1**(a) [*P*-1] with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1A and H2A, to emphasize valency of the nitrogen atoms.

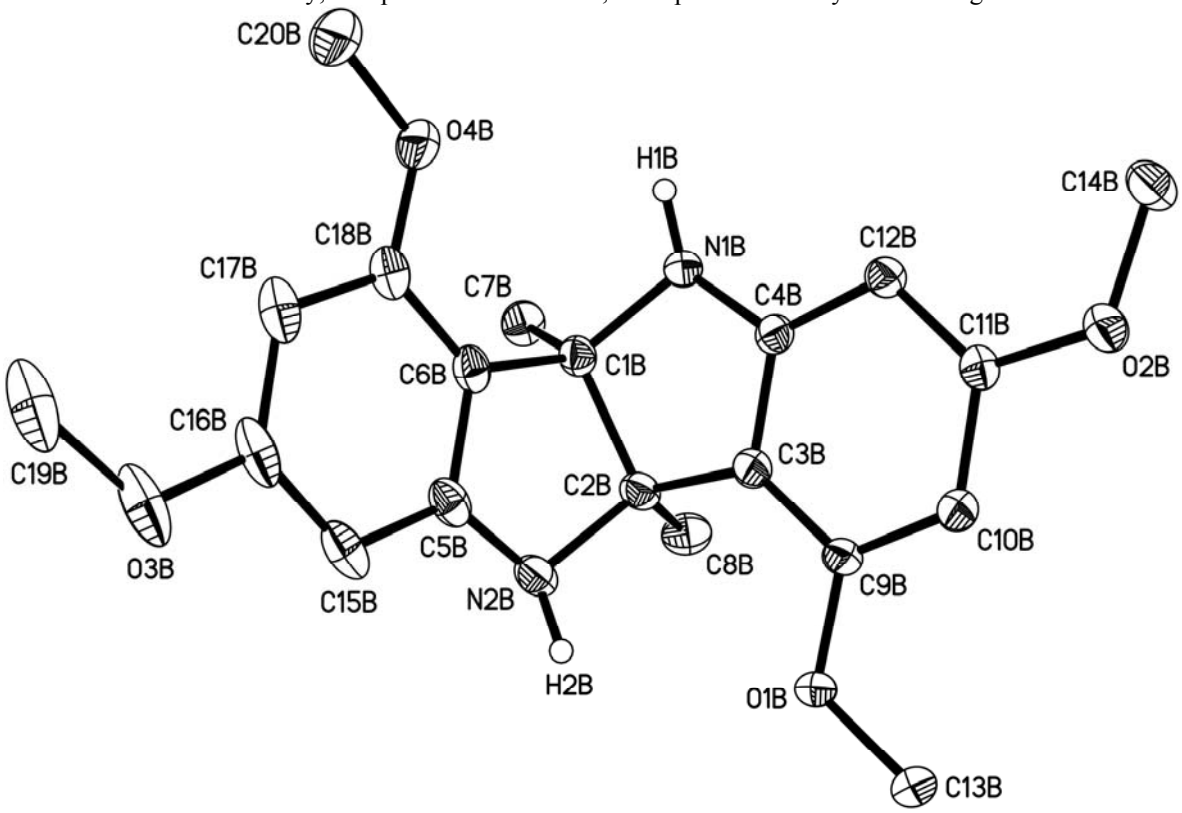


Figure S50. ORTEP diagram of **1**(b) [*P*-1] with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1B and H2B, to emphasize valency of the nitrogen atoms.

Experimental Details for the Structure Determination of (+/-)-**1** [*P*-1].

Data collection

A crystal (0.48 x 0.28 x 0.14 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(1) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 10 seconds and a detector distance of 5.04 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -33º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 3974 strong reflections from the actual data collection after integration.[3] See Table S8 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group *P*-1 was determined based on the lack of systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were found from the difference Fourier map; their positions and isotropic displacement parameters were refined independently from those of all other atoms. The final full matrix least squares refinement converged to $R1 = 0.0429$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1208$ (F^2, all data).

Structure description

The structure is the one suggested. There are two independent molecules in the asymmetric unit with all atoms in general positions. Weak to moderate intermolecular and intramolecular hydrogen bonding is observed (see Table S14).

References

[1] *APEX2* V2.1-0, Bruker Analytical X-ray Systems, Madison, WI (2006).

[2] *SADABS* 2004/1, An empirical correction for absorption anisotropy, R. Blessing, *Acta Cryst.* **A51**, 33-38 (1995).

[3] *SAINT* V7.34A, Bruker Analytical X-ray Systems, Madison, WI (2006).

[4] *SIR97, A new tool for crystal structure determination and refinement,* A. Altomare, M. C. Burla, M. Camalli, G. L. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori, R. Spagna. *J. Appl. Cryst.* **32**, 115-119 (1999).

[5] *SHELXTL* V6.14, Bruker Analytical X-ray Systems, Madison, WI (2000).

Some equations of interest:

$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| / \Sigma|F_o^2|$

$R1 = \Sigma||F_o| - |F_c|| / \Sigma|F_o|$

$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$

where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$P = 1/3 \max(0, F_o^2) + 2/3\, F_c^2$

$GooF = S = [\Sigma[w(F_o^2 - F_c^2)^2] / (m-n)]^{1/2}$

where m = number of reflections and n = number of parameters

Table S8. Crystal data and structure refinement for (+/-)-**1** [*P*-1].

Identification code	(+/-)-**1**, JONJK13	
Empirical formula	C20 H24 N2 O4	
Formula weight	356.41	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	*a* = 10.5300(12) Å	α = 99.573(2)°
	b = 12.5823(15) Å	β = 108.832(2)°
	c = 15.1214(18) Å	γ = 97.267(2)°
Volume	1834.6(4) Å^3	
Z	4	
Density (calculated)	1.290 Mg/m^3	
Absorption coefficient	0.090 mm^{-1}	
F(000)	760	
Crystal color, morphology	colorless, block	
Crystal size	0.48 x 0.28 x 0.14 mm^3	
Theta range for data collection	1.67 to 31.51°	
Index ranges	$-15 \le h \le 15$, $-18 \le k \le 18$, $-22 \le l \le 22$	
Reflections collected	31906	
Independent reflections	12131 [*R*(int) = 0.0297]	
Observed reflections	9371	
Completeness to theta = 31.51°	99.1%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9875 and 0.9579	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12131 / 0 / 661	
Goodness-of-fit on F^2	1.033	
Final *R* indices [*I*>2sigma(*I*)]	*R*1 = 0.0429, *wR*2 = 0.1102	
R indices (all data)	*R*1 = 0.0583, *wR*2 = 0.1208	
Largest diff. peak and hole	0.380 and -0.372 e.Å^{-3}	

Table S9. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for (+/-)-**1** [*P*-1]. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1A	7502(1)	2801(1)	7928(1)	21(1)
O2A	7144(1)	-9(1)	5274(1)	18(1)
O3A	396(1)	2993(1)	6358(1)	22(1)
O4A	1315(1)	-578(1)	6694(1)	21(1)
N1A	4442(1)	-446(1)	7357(1)	16(1)
N2A	4812(1)	2373(1)	8418(1)	17(1)
C1A	4113(1)	407(1)	8011(1)	16(1)
C2A	5403(1)	1368(1)	8367(1)	15(1)
C3A	5948(1)	1136(1)	7555(1)	14(1)
C4A	5361(1)	86(1)	6998(1)	14(1)
C5A	3470(1)	2109(1)	7762(1)	16(1)
C6A	3006(1)	982(1)	7510(1)	15(1)
C7A	3778(1)	-77(1)	8787(1)	21(1)
C8A	6479(1)	1365(1)	9324(1)	20(1)
C9A	6940(1)	1776(1)	7338(1)	15(1)
C10A	7302(1)	1372(1)	6558(1)	16(1)
C11A	6699(1)	300(1)	6016(1)	15(1)
C12A	5730(1)	-370(1)	6229(1)	15(1)
C13A	8602(1)	3413(1)	7749(1)	33(1)
C14A	6669(1)	-1131(1)	4762(1)	19(1)
C15A	2643(1)	2838(1)	7407(1)	18(1)
C16A	1314(1)	2376(1)	6782(1)	18(1)
C17A	801(1)	1239(1)	6526(1)	18(1)
C18A	1664(1)	544(1)	6901(1)	16(1)
C19A	857(1)	4154(1)	6609(1)	27(1)
C20A	-92(1)	-1060(1)	6178(1)	26(1)
O1B	670(1)	4071(1)	835(1)	22(1)
O2B	-322(1)	475(1)	1351(1)	25(1)
O3B	6091(1)	6523(1)	5148(1)	38(1)
O4B	6713(1)	3025(1)	3578(1)	28(1)
N1B	4270(1)	2214(1)	1603(1)	19(1)
N2B	3883(1)	5002(1)	1752(1)	22(1)
C1B	4863(1)	3373(1)	1720(1)	18(1)

C2B	3592(1)	3863(1)	1182(1)	19(1)
C3B	2439(1)	3078(1)	1269(1)	17(1)
C4B	2896(1)	2133(1)	1497(1)	17(1)
C5B	4695(1)	5014(1)	2694(1)	21(1)
C6B	5288(1)	4081(1)	2725(1)	20(1)
C7B	6038(1)	3473(1)	1338(1)	24(1)
C8B	3376(1)	3858(1)	133(1)	25(1)
C9B	1063(1)	3123(1)	1078(1)	18(1)
C10B	168(1)	2243(1)	1130(1)	20(1)
C11B	660(1)	1298(1)	1351(1)	19(1)
C12B	2028(1)	1225(1)	1547(1)	18(1)
C13B	-748(1)	4108(1)	577(1)	29(1)
C14B	11(1)	-589(1)	1360(1)	26(1)
C15B	4967(1)	5836(1)	3506(1)	26(1)
C16B	5861(1)	5695(1)	4367(1)	28(1)
C17B	6469(1)	4773(1)	4430(1)	27(1)
C18B	6173(1)	3962(1)	3593(1)	23(1)
C19B	7087(2)	6487(1)	6034(1)	43(1)
C20B	7587(2)	2868(1)	4467(1)	46(1)

Table S10. Bond lengths [Å] and angles [°] for (+/-)-**1** [*P*-1].

O(1A)-C(9A)	1.3759(12)	C(12A)-H(12A)	0.976(13)
O(1A)-C(13A)	1.4327(14)	C(13A)-H(13A)	0.972(17)
O(2A)-C(11A)	1.3671(12)	C(13A)-H(13B)	0.969(17)
O(2A)-C(14A)	1.4322(12)	C(13A)-H(13C)	1.042(17)
O(3A)-C(16A)	1.3793(12)	C(14A)-H(14A)	0.981(14)
O(3A)-C(19A)	1.4251(14)	C(14A)-H(14B)	0.994(15)
O(4A)-C(18A)	1.3699(12)	C(14A)-H(14C)	0.982(14)
O(4A)-C(20A)	1.4287(13)	C(15A)-C(16A)	1.3955(14)
N(1A)-C(4A)	1.4058(12)	C(15A)-H(15A)	0.969(14)
N(1A)-C(1A)	1.4873(13)	C(16A)-C(17A)	1.4044(15)
N(1A)-H(1A)	0.895(14)	C(17A)-C(18A)	1.3948(14)
N(2A)-C(5A)	1.3984(13)	C(17A)-H(17A)	0.953(15)
N(2A)-C(2A)	1.4806(13)	C(19A)-H(19A)	0.973(15)
N(2A)-H(2A)	0.896(15)	C(19A)-H(19B)	0.992(15)
C(1A)-C(6A)	1.5104(14)	C(19A)-H(19C)	0.983(16)
C(1A)-C(7A)	1.5239(14)	C(20A)-H(20A)	1.001(17)
C(1A)-C(2A)	1.5790(14)	C(20A)-H(20B)	0.958(17)
C(2A)-C(3A)	1.5159(13)	C(20A)-H(20C)	0.989(16)
C(2A)-C(8A)	1.5231(14)	O(1B)-C(9B)	1.3785(12)
C(3A)-C(4A)	1.3877(13)	O(1B)-C(13B)	1.4255(14)
C(3A)-C(9A)	1.3946(13)	O(2B)-C(11B)	1.3673(13)
C(4A)-C(12A)	1.3992(14)	O(2B)-C(14B)	1.4274(14)
C(5A)-C(6A)	1.3865(14)	O(3B)-C(16B)	1.3715(14)
C(5A)-C(15A)	1.3976(14)	O(3B)-C(19B)	1.422(2)
C(6A)-C(18A)	1.3933(13)	O(4B)-C(18B)	1.3726(14)
C(7A)-H(7A)	0.998(14)	O(4B)-C(20B)	1.4234(16)
C(7A)-H(7B)	0.990(15)	N(1B)-C(4B)	1.3918(13)
C(7A)-H(7C)	0.969(16)	N(1B)-C(1B)	1.4696(13)
C(8A)-H(8A)	0.961(16)	N(1B)-H(1B)	0.870(17)
C(8A)-H(8B)	0.990(15)	N(2B)-C(5B)	1.4020(15)
C(8A)-H(8C)	1.002(15)	N(2B)-C(2B)	1.4825(14)
C(9A)-C(10A)	1.3905(14)	N(2B)-H(2B)	0.882(17)
C(10A)-C(11A)	1.4052(14)	C(1B)-C(6B)	1.5207(15)
C(10A)-H(10A)	0.959(15)	C(1B)-C(7B)	1.5253(15)
C(11A)-C(12A)	1.3936(14)	C(1B)-C(2B)	1.5828(15)

C(2B)-C(3B)	1.5207(14)	C(11A)-O(2A)-C(14A)	116.25(8)
C(2B)-C(8B)	1.5266(16)	C(16A)-O(3A)-C(19A)	116.53(9)
C(3B)-C(4B)	1.3924(14)	C(18A)-O(4A)-C(20A)	117.22(9)
C(3B)-C(9B)	1.3932(14)	C(4A)-N(1A)-C(1A)	107.82(8)
C(4B)-C(12B)	1.3989(14)	C(4A)-N(1A)-H(1A)	115.0(9)
C(5B)-C(15B)	1.3910(15)	C(1A)-N(1A)-H(1A)	114.1(9)
C(5B)-C(6B)	1.3978(15)	C(5A)-N(2A)-C(2A)	107.77(8)
C(6B)-C(18B)	1.3878(16)	C(5A)-N(2A)-H(2A)	115.4(9)
C(7B)-H(7D)	1.003(15)	C(2A)-N(2A)-H(2A)	117.9(9)
C(7B)-H(7E)	0.983(15)	N(1A)-C(1A)-C(6A)	114.28(8)
C(7B)-H(7F)	1.001(15)	N(1A)-C(1A)-C(7A)	110.30(8)
C(8B)-H(8D)	1.007(16)	C(6A)-C(1A)-C(7A)	111.38(8)
C(8B)-H(8E)	0.979(17)	N(1A)-C(1A)-C(2A)	103.76(7)
C(8B)-H(8F)	0.988(17)	C(6A)-C(1A)-C(2A)	100.63(8)
C(9B)-C(10B)	1.3906(15)	C(7A)-C(1A)-C(2A)	116.12(8)
C(10B)-C(11B)	1.4037(15)	N(2A)-C(2A)-C(3A)	114.53(8)
C(10B)-H(10B)	0.981(15)	N(2A)-C(2A)-C(8A)	111.37(8)
C(11B)-C(12B)	1.3926(15)	C(3A)-C(2A)-C(8A)	111.13(8)
C(12B)-H(12B)	0.958(14)	N(2A)-C(2A)-C(1A)	103.59(7)
C(13B)-H(13D)	0.968(17)	C(3A)-C(2A)-C(1A)	100.61(7)
C(13B)-H(13E)	0.965(16)	C(8A)-C(2A)-C(1A)	115.09(8)
C(13B)-H(13F)	0.992(16)	C(4A)-C(3A)-C(9A)	118.87(9)
C(14B)-H(14D)	0.967(16)	C(4A)-C(3A)-C(2A)	110.57(8)
C(14B)-H(14E)	0.977(17)	C(9A)-C(3A)-C(2A)	130.48(9)
C(14B)-H(14F)	0.993(16)	C(3A)-C(4A)-C(12A)	122.98(9)
C(15B)-C(16B)	1.3931(19)	C(3A)-C(4A)-N(1A)	110.42(8)
C(15B)-H(15B)	0.953(16)	C(12A)-C(4A)-N(1A)	126.48(9)
C(16B)-C(17B)	1.3966(19)	C(6A)-C(5A)-C(15A)	122.13(9)
C(17B)-C(18B)	1.4047(16)	C(6A)-C(5A)-N(2A)	110.55(9)
C(17B)-H(17B)	0.967(16)	C(15A)-C(5A)-N(2A)	127.28(9)
C(19B)-H(19D)	0.958(18)	C(5A)-C(6A)-C(18A)	119.91(9)
C(19B)-H(19E)	0.981(17)	C(5A)-C(6A)-C(1A)	110.34(8)
C(19B)-H(19F)	1.005(19)	C(18A)-C(6A)-C(1A)	129.71(9)
C(20B)-H(20D)	0.944(19)	C(1A)-C(7A)-H(7A)	109.6(8)
C(20B)-H(20E)	0.948(19)	C(1A)-C(7A)-H(7B)	110.4(8)
C(20B)-H(20F)	1.02(2)	H(7A)-C(7A)-H(7B)	109.4(11)
C(9A)-O(1A)-C(13A)	115.62(8)	C(1A)-C(7A)-H(7C)	109.7(9)

H(7A)-C(7A)-H(7C)	108.6(12)	C(15A)-C(16A)-C(17A)	122.71(9)
H(7B)-C(7A)-H(7C)	109.3(12)	C(18A)-C(17A)-C(16A)	118.52(9)
C(2A)-C(8A)-H(8A)	108.9(9)	C(18A)-C(17A)-H(17A)	122.3(9)
C(2A)-C(8A)-H(8B)	111.7(9)	C(16A)-C(17A)-H(17A)	119.2(9)
H(8A)-C(8A)-H(8B)	110.0(13)	O(4A)-C(18A)-C(6A)	115.09(9)
C(2A)-C(8A)-H(8C)	111.0(9)	O(4A)-C(18A)-C(17A)	124.85(9)
H(8A)-C(8A)-H(8C)	109.2(13)	C(6A)-C(18A)-C(17A)	120.02(9)
H(8B)-C(8A)-H(8C)	105.9(12)	O(3A)-C(19A)-H(19A)	104.2(9)
O(1A)-C(9A)-C(10A)	123.69(9)	O(3A)-C(19A)-H(19B)	112.0(8)
O(1A)-C(9A)-C(3A)	116.13(9)	H(19A)-C(19A)-H(19B)	111.5(12)
C(10A)-C(9A)-C(3A)	120.17(9)	O(3A)-C(19A)-H(19C)	111.4(9)
C(9A)-C(10A)-C(11A)	119.40(9)	H(19A)-C(19A)-H(19C)	109.2(13)
C(9A)-C(10A)-H(10A)	123.1(9)	H(19B)-C(19A)-H(19C)	108.6(13)
C(11A)-C(10A)-H(10A)	117.5(9)	O(4A)-C(20A)-H(20A)	106.1(9)
O(2A)-C(11A)-C(12A)	124.03(9)	O(4A)-C(20A)-H(20B)	111.8(10)
O(2A)-C(11A)-C(10A)	114.14(8)	H(20A)-C(20A)-H(20B)	112.0(14)
C(12A)-C(11A)-C(10A)	121.83(9)	O(4A)-C(20A)-H(20C)	110.7(9)
C(11A)-C(12A)-C(4A)	116.68(9)	H(20A)-C(20A)-H(20C)	110.0(13)
C(11A)-C(12A)-H(12A)	122.9(8)	H(20B)-C(20A)-H(20C)	106.3(13)
C(4A)-C(12A)-H(12A)	120.4(8)	C(9B)-O(1B)-C(13B)	117.11(9)
O(1A)-C(13A)-H(13A)	104.0(10)	C(11B)-O(2B)-C(14B)	117.55(9)
O(1A)-C(13A)-H(13B)	109.3(10)	C(16B)-O(3B)-C(19B)	118.26(12)
H(13A)-C(13A)-H(13B)	111.3(14)	C(18B)-O(4B)-C(20B)	117.42(10)
O(1A)-C(13A)-H(13C)	109.8(9)	C(4B)-N(1B)-C(1B)	108.49(8)
H(13A)-C(13A)-H(13C)	107.6(13)	C(4B)-N(1B)-H(1B)	116.7(11)
H(13B)-C(13A)-H(13C)	114.3(14)	C(1B)-N(1B)-H(1B)	117.5(11)
O(2A)-C(14A)-H(14A)	105.8(8)	C(5B)-N(2B)-C(2B)	107.77(9)
O(2A)-C(14A)-H(14B)	108.9(8)	C(5B)-N(2B)-H(2B)	115.4(11)
H(14A)-C(14A)-H(14B)	110.2(11)	C(2B)-N(2B)-H(2B)	114.8(11)
O(2A)-C(14A)-H(14C)	113.2(8)	N(1B)-C(1B)-C(6B)	115.65(9)
H(14A)-C(14A)-H(14C)	109.4(11)	N(1B)-C(1B)-C(7B)	110.81(9)
H(14B)-C(14A)-H(14C)	109.3(12)	C(6B)-C(1B)-C(7B)	110.93(9)
C(16A)-C(15A)-C(5A)	116.68(9)	N(1B)-C(1B)-C(2B)	103.37(8)
C(16A)-C(15A)-H(15A)	122.3(8)	C(6B)-C(1B)-C(2B)	100.35(8)
C(5A)-C(15A)-H(15A)	121.0(8)	C(7B)-C(1B)-C(2B)	115.30(9)
O(3A)-C(16A)-C(15A)	123.01(9)	N(2B)-C(2B)-C(3B)	115.77(8)
O(3A)-C(16A)-C(17A)	114.29(9)	N(2B)-C(2B)-C(8B)	110.85(9)

C(3B)-C(2B)-C(8B)	110.90(9)	C(11B)-C(12B)-C(4B)	116.88(9)
N(2B)-C(2B)-C(1B)	103.55(8)	C(11B)-C(12B)-H(12B)	121.9(8)
C(3B)-C(2B)-C(1B)	100.21(8)	C(4B)-C(12B)-H(12B)	121.2(8)
C(8B)-C(2B)-C(1B)	115.12(9)	O(1B)-C(13B)-H(13D)	104.9(10)
C(4B)-C(3B)-C(9B)	118.98(9)	O(1B)-C(13B)-H(13E)	113.1(9)
C(4B)-C(3B)-C(2B)	109.59(9)	H(13D)-C(13B)-H(13E)	111.4(13)
C(9B)-C(3B)-C(2B)	131.12(9)	O(1B)-C(13B)-H(13F)	109.3(9)
N(1B)-C(4B)-C(3B)	110.60(9)	H(13D)-C(13B)-H(13F)	109.3(13)
N(1B)-C(4B)-C(12B)	126.75(9)	H(13E)-C(13B)-H(13F)	108.8(13)
C(3B)-C(4B)-C(12B)	122.55(9)	O(2B)-C(14B)-H(14D)	105.0(9)
C(15B)-C(5B)-C(6B)	122.23(11)	O(2B)-C(14B)-H(14E)	110.7(9)
C(15B)-C(5B)-N(2B)	127.16(11)	H(14D)-C(14B)-H(14E)	110.8(13)
C(6B)-C(5B)-N(2B)	110.54(9)	O(2B)-C(14B)-H(14F)	110.9(9)
C(18B)-C(6B)-C(5B)	119.25(10)	H(14D)-C(14B)-H(14F)	112.1(13)
C(18B)-C(6B)-C(1B)	130.82(10)	H(14E)-C(14B)-H(14F)	107.4(13)
C(5B)-C(6B)-C(1B)	109.84(9)	C(5B)-C(15B)-C(16B)	117.25(11)
C(1B)-C(7B)-H(7D)	108.8(9)	C(5B)-C(15B)-H(15B)	123.9(10)
C(1B)-C(7B)-H(7E)	111.6(8)	C(16B)-C(15B)-H(15B)	118.8(9)
H(7D)-C(7B)-H(7E)	108.1(12)	O(3B)-C(16B)-C(15B)	115.11(12)
C(1B)-C(7B)-H(7F)	109.7(9)	O(3B)-C(16B)-C(17B)	122.58(12)
H(7D)-C(7B)-H(7F)	108.7(12)	C(15B)-C(16B)-C(17B)	122.30(11)
H(7E)-C(7B)-H(7F)	109.9(12)	C(16B)-C(17B)-C(18B)	118.79(11)
C(2B)-C(8B)-H(8D)	109.4(9)	C(16B)-C(17B)-H(17B)	121.0(10)
C(2B)-C(8B)-H(8E)	110.6(9)	C(18B)-C(17B)-H(17B)	120.2(10)
H(8D)-C(8B)-H(8E)	110.3(13)	O(4B)-C(18B)-C(6B)	116.48(10)
C(2B)-C(8B)-H(8F)	111.2(9)	O(4B)-C(18B)-C(17B)	123.35(11)
H(8D)-C(8B)-H(8F)	108.2(13)	C(6B)-C(18B)-C(17B)	120.17(11)
H(8E)-C(8B)-H(8F)	107.1(13)	O(3B)-C(19B)-H(19D)	104.9(11)
O(1B)-C(9B)-C(10B)	123.54(9)	O(3B)-C(19B)-H(19E)	111.7(10)
O(1B)-C(9B)-C(3B)	116.10(9)	H(19D)-C(19B)-H(19E)	110.0(14)
C(10B)-C(9B)-C(3B)	120.36(9)	O(3B)-C(19B)-H(19F)	112.1(10)
C(9B)-C(10B)-C(11B)	119.14(10)	H(19D)-C(19B)-H(19F)	110.8(15)
C(9B)-C(10B)-H(10B)	122.7(9)	H(19E)-C(19B)-H(19F)	107.5(14)
C(11B)-C(10B)-H(10B)	118.1(9)	O(4B)-C(20B)-H(20D)	107.6(11)
O(2B)-C(11B)-C(12B)	124.07(9)	O(4B)-C(20B)-H(20E)	111.2(11)
O(2B)-C(11B)-C(10B)	113.87(9)	H(20D)-C(20B)-H(20E)	112.9(16)
C(12B)-C(11B)-C(10B)	122.06(10)	O(4B)-C(20B)-H(20F)	108.5(11)

H(20D)-C(20B)-H(20F)	106.3(15)	H(20E)-C(20B)-H(20F)	110.1(15)

Table S11. Anisotropic displacement parameters ($Å^2$ x 10^3) for (+/-)-**1** [*P*-1]. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$h^2 a^{*2} U_{11}$ + ... + 2 h k a* b* U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1A	18(1)	15(1)	27(1)	-5(1)	11(1)	-3(1)
O2A	20(1)	16(1)	17(1)	0(1)	9(1)	3(1)
O3A	18(1)	21(1)	27(1)	6(1)	6(1)	6(1)
O4A	15(1)	17(1)	26(1)	3(1)	3(1)	-1(1)
N1A	14(1)	15(1)	17(1)	2(1)	5(1)	0(1)
N2A	14(1)	15(1)	18(1)	0(1)	4(1)	1(1)
C1A	14(1)	17(1)	15(1)	3(1)	5(1)	1(1)
C2A	13(1)	16(1)	15(1)	0(1)	4(1)	2(1)
C3A	13(1)	15(1)	14(1)	2(1)	4(1)	2(1)
C4A	13(1)	14(1)	15(1)	4(1)	3(1)	2(1)
C5A	14(1)	19(1)	15(1)	1(1)	6(1)	2(1)
C6A	13(1)	18(1)	15(1)	3(1)	6(1)	2(1)
C7A	19(1)	26(1)	19(1)	8(1)	8(1)	2(1)
C8A	17(1)	24(1)	15(1)	2(1)	3(1)	2(1)
C9A	13(1)	12(1)	18(1)	0(1)	5(1)	1(1)
C10A	15(1)	14(1)	19(1)	2(1)	7(1)	2(1)

C11A	15(1)	16(1)	14(1)	3(1)	5(1)	5(1)
C12A	15(1)	13(1)	16(1)	1(1)	4(1)	2(1)
C13A	27(1)	20(1)	48(1)	-10(1)	23(1)	-8(1)
C14A	20(1)	17(1)	17(1)	-1(1)	6(1)	4(1)
C15A	17(1)	17(1)	20(1)	2(1)	8(1)	2(1)
C16A	16(1)	21(1)	18(1)	5(1)	8(1)	5(1)
C17A	13(1)	22(1)	18(1)	3(1)	5(1)	2(1)
C18A	15(1)	17(1)	16(1)	3(1)	6(1)	0(1)
C19A	23(1)	21(1)	37(1)	10(1)	8(1)	5(1)
C20A	16(1)	23(1)	30(1)	3(1)	2(1)	-4(1)
O1B	17(1)	15(1)	32(1)	5(1)	6(1)	6(1)
O2B	21(1)	18(1)	36(1)	8(1)	10(1)	2(1)
O3B	56(1)	23(1)	30(1)	-8(1)	23(1)	-11(1)
O4B	27(1)	27(1)	23(1)	0(1)	0(1)	5(1)
N1B	16(1)	16(1)	22(1)	3(1)	5(1)	5(1)
N2B	22(1)	15(1)	30(1)	3(1)	12(1)	4(1)
C1B	17(1)	17(1)	20(1)	1(1)	7(1)	3(1)
C2B	20(1)	16(1)	23(1)	3(1)	9(1)	4(1)
C3B	18(1)	16(1)	18(1)	2(1)	6(1)	4(1)
C4B	17(1)	17(1)	15(1)	1(1)	4(1)	4(1)
C5B	21(1)	16(1)	28(1)	2(1)	14(1)	-1(1)
C6B	18(1)	18(1)	22(1)	0(1)	9(1)	-1(1)
C7B	20(1)	25(1)	27(1)	3(1)	12(1)	5(1)
C8B	27(1)	28(1)	25(1)	10(1)	12(1)	10(1)
C9B	18(1)	14(1)	19(1)	2(1)	5(1)	4(1)
C10B	16(1)	18(1)	22(1)	3(1)	5(1)	3(1)
C11B	19(1)	17(1)	18(1)	2(1)	6(1)	1(1)
C12B	20(1)	16(1)	18(1)	3(1)	5(1)	4(1)
C13B	18(1)	21(1)	43(1)	7(1)	4(1)	6(1)
C14B	29(1)	18(1)	32(1)	7(1)	12(1)	2(1)
C15B	32(1)	16(1)	32(1)	-2(1)	19(1)	-3(1)
C16B	36(1)	19(1)	27(1)	-5(1)	19(1)	-10(1)
C17B	30(1)	24(1)	21(1)	-1(1)	10(1)	-8(1)
C18B	22(1)	20(1)	23(1)	0(1)	8(1)	-2(1)
C19B	62(1)	30(1)	27(1)	-6(1)	22(1)	-20(1)
C20B	53(1)	35(1)	28(1)	0(1)	-12(1)	9(1)

Table S12. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for (+/-)-**1** [*P*-1].

	x	y	z	$U_{(eq)}$
H1A	3708(15)	-899(12)	6912(10)	22(3)
H2A	5312(15)	2999(12)	8407(10)	25(3)
H7A	2899(14)	-621(12)	8494(10)	23(3)
H7B	4516(15)	-444(12)	9113(10)	25(4)
H7C	3676(15)	503(13)	9254(11)	29(4)
H8A	7256(16)	1935(13)	9450(11)	33(4)
H8B	6117(15)	1477(12)	9853(11)	28(4)
H8C	6779(15)	640(13)	9314(11)	30(4)
H10A	7957(15)	1791(12)	6369(11)	29(4)
H12A	5300(13)	-1116(11)	5859(9)	16(3)
H13A	8864(17)	4099(14)	8225(12)	41(4)
H13B	9346(17)	3013(13)	7838(12)	38(4)
H13C	8240(17)	3597(13)	7076(13)	40(4)
H14A	7113(13)	-1223(11)	4282(10)	18(3)
H14B	6960(15)	-1616(12)	5216(11)	26(4)
H14C	5672(15)	-1323(11)	4439(10)	23(3)
H15A	2990(14)	3623(12)	7582(10)	23(3)
H17A	-115(15)	967(12)	6094(11)	27(4)
H19A	75(16)	4437(12)	6254(11)	29(4)
H19B	1143(15)	4450(12)	7308(11)	27(4)
H19C	1628(16)	4367(13)	6401(12)	34(4)
H20A	-162(17)	-1871(14)	6122(12)	38(4)
H20B	-675(16)	-773(13)	6490(12)	35(4)
H20C	-390(15)	-896(12)	5533(12)	30(4)
H1B	4739(18)	1847(14)	1987(13)	41(4)
H2B	3158(17)	5307(13)	1691(12)	34(4)
H7D	6788(15)	3147(12)	1731(11)	29(4)
H7E	5749(15)	3075(12)	667(11)	26(4)
H7F	6402(15)	4265(13)	1398(11)	30(4)
H8D	2502(16)	4119(13)	-164(11)	34(4)
H8E	4150(17)	4328(13)	78(11)	36(4)
H8F	3302(16)	3111(14)	-233(12)	36(4)
H10B	-807(16)	2245(12)	1001(11)	30(4)

H12B	2364(14)	582(11)	1704(10)	22(3)
H13D	-814(17)	4836(14)	459(12)	38(4)
H13E	-1317(16)	3545(13)	24(11)	31(4)
H13F	-1045(16)	4023(12)	1126(11)	31(4)
H14D	-831(17)	-1063(13)	1289(11)	34(4)
H14E	323(16)	-831(13)	831(12)	38(4)
H14F	760(16)	-582(12)	1964(11)	31(4)
H15B	4601(16)	6490(13)	3501(11)	32(4)
H17B	7077(16)	4686(13)	5036(12)	34(4)
H19D	7118(18)	7146(15)	6467(13)	43(5)
H19E	6827(17)	5843(14)	6270(12)	41(4)
H19F	8010(19)	6459(14)	5980(13)	46(5)
H20D	7793(19)	2160(16)	4355(13)	50(5)
H20E	8379(19)	3432(16)	4738(14)	49(5)
H20F	7051(19)	2861(15)	4924(14)	50(5)

Table S13. Torsion angles [°] for (+/-)-**1** [*P*-1].

C4A-N1A-C1A-C6A	82.89(10)	C2A-C1A-C6A-C5A	-14.72(10)
C4A-N1A-C1A-C7A	-150.72(8)	N1A-C1A-C6A-C18A	57.35(13)
C4A-N1A-C1A-C2A	-25.70(10)	C7A-C1A-C6A-C18A	-68.47(13)
C5A-N2A-C2A-C3A	82.10(10)	C2A-C1A-C6A-C18A	167.85(10)
C5A-N2A-C2A-C8A	-150.73(8)	C13A-O1A-C9A-C10A	-5.70(15)
C5A-N2A-C2A-C1A	-26.46(10)	C13A-O1A-C9A-C3A	174.89(10)
N1A-C1A-C2A-N2A	142.59(8)	C4A-C3A-C9A-O1A	-178.75(9)
C6A-C1A-C2A-N2A	24.12(9)	C2A-C3A-C9A-O1A	-2.36(15)
C7A-C1A-C2A-N2A	-96.22(10)	C4A-C3A-C9A-C10A	1.82(14)
N1A-C1A-C2A-C3A	23.92(9)	C2A-C3A-C9A-C10A	178.21(9)
C6A-C1A-C2A-C3A	-94.56(8)	O1A-C9A-C10A-C11A	178.09(9)
C7A-C1A-C2A-C3A	145.10(9)	C3A-C9A-C10A-C11A	-2.52(15)
N1A-C1A-C2A-C8A	-95.60(9)	C14A-O2A-C11A-C12A	-6.52(13)
C6A-C1A-C2A-C8A	145.93(8)	C14A-O2A-C11A-C10A	173.65(8)
C7A-C1A-C2A-C8A	25.59(12)	C9A-C10A-C11A-O2A	-179.36(9)
N2A-C2A-C3A-C4A	-125.58(9)	C9A-C10A-C11A-C12A	0.80(15)
C8A-C2A-C3A-C4A	107.14(9)	O2A-C11A-C12A-C4A	-178.28(9)
C1A-C2A-C3A-C4A	-15.20(10)	C10A-C11A-C12A-C4A	1.53(14)
N2A-C2A-C3A-C9A	57.80(13)	C3A-C4A-C12A-C11A	-2.28(14)
C8A-C2A-C3A-C9A	-69.49(13)	N1A-C4A-C12A-C11A	-177.94(9)
C1A-C2A-C3A-C9A	168.17(10)	C6A-C5A-C15A-C16A	0.10(14)
C9A-C3A-C4A-C12A	0.65(14)	N2A-C5A-C15A-C16A	-177.70(9)
C2A-C3A-C4A-C12A	-176.42(9)	C19A-O3A-C16A-C15A	-1.48(14)
C9A-C3A-C4A-N1A	176.93(8)	C19A-O3A-C16A-C17A	178.85(10)
C2A-C3A-C4A-N1A	-0.14(11)	C5A-C15A-C16A-O3A	-178.42(9)
C1A-N1A-C4A-C3A	16.94(10)	C5A-C15A-C16A-C17A	1.22(15)
C1A-N1A-C4A-C12A	-166.95(9)	O3A-C16A-C17A-C18A	178.60(9)
C2A-N2A-C5A-C6A	18.11(11)	C15A-C16A-C17A-C18A	-1.07(15)
C2A-N2A-C5A-C15A	-163.88(9)	C20A-O4A-C18A-C6A	171.95(9)
C15A-C5A-C6A-C18A	-1.55(15)	C20A-O4A-C18A-C17A	-10.25(15)
N2A-C5A-C6A-C18A	176.58(9)	C5A-C6A-C18A-O4A	179.61(9)
C15A-C5A-C6A-C1A	-179.27(9)	C1A-C6A-C18A-O4A	-3.17(15)
N2A-C5A-C6A-C1A	-1.14(11)	C5A-C6A-C18A-C17A	1.70(15)
N1A-C1A-C6A-C5A	-125.22(9)	C1A-C6A-C18A-C17A	178.92(9)
C7A-C1A-C6A-C5A	108.96(10)	C16A-C17A-C18A-O4A	-178.12(9)

C16A-C17A-C18A-C6A	-0.42(15)	C2B-C1B-C6B-C18B	166.64(11)
C4B-N1B-C1B-C6B	81.76(11)	N1B-C1B-C6B-C5B	-127.33(9)
C4B-N1B-C1B-C7B	-150.91(9)	C7B-C1B-C6B-C5B	105.39(10)
C4B-N1B-C1B-C2B	-26.84(10)	C2B-C1B-C6B-C5B	-16.94(10)
C5B-N2B-C2B-C3B	81.08(11)	C13B-O1B-C9B-C10B	-3.42(15)
C5B-N2B-C2B-C8B	-151.50(9)	C13B-O1B-C9B-C3B	176.13(10)
C5B-N2B-C2B-C1B	-27.51(10)	C4B-C3B-C9B-O1B	-178.46(9)
N1B-C1B-C2B-N2B	145.76(8)	C2B-C3B-C9B-O1B	-5.60(16)
C6B-C1B-C2B-N2B	26.04(10)	C4B-C3B-C9B-C10B	1.10(15)
C7B-C1B-C2B-N2B	-93.16(10)	C2B-C3B-C9B-C10B	173.96(10)
N1B-C1B-C2B-C3B	25.90(9)	O1B-C9B-C10B-C11B	177.70(9)
C6B-C1B-C2B-C3B	-93.81(9)	C3B-C9B-C10B-C11B	-1.83(16)
C7B-C1B-C2B-C3B	146.99(9)	C14B-O2B-C11B-C12B	-13.14(15)
N1B-C1B-C2B-C8B	-93.09(10)	C14B-O2B-C11B-C10B	166.71(10)
C6B-C1B-C2B-C8B	147.20(9)	C9B-C10B-C11B-O2B	-177.93(9)
C7B-C1B-C2B-C8B	27.99(13)	C9B-C10B-C11B-C12B	1.92(16)
N2B-C2B-C3B-C4B	-128.10(10)	O2B-C11B-C12B-C4B	178.62(9)
C8B-C2B-C3B-C4B	104.50(10)	C10B-C11B-C12B-C4B	-1.22(15)
C1B-C2B-C3B-C4B	-17.54(10)	N1B-C4B-C12B-C11B	-175.57(10)
N2B-C2B-C3B-C9B	58.54(15)	C3B-C4B-C12B-C11B	0.47(15)
C8B-C2B-C3B-C9B	-68.86(14)	C6B-C5B-C15B-C16B	-0.21(16)
C1B-C2B-C3B-C9B	169.10(11)	N2B-C5B-C15B-C16B	-176.98(10)
C1B-N1B-C4B-C3B	16.52(11)	C19B-O3B-C16B-C15B	-174.31(10)
C1B-N1B-C4B-C12B	-167.05(10)	C19B-O3B-C16B-C17B	6.20(17)
C9B-C3B-C4B-N1B	176.19(9)	C5B-C15B-C16B-O3B	-179.63(10)
C2B-C3B-C4B-N1B	1.90(12)	C5B-C15B-C16B-C17B	-0.14(17)
C9B-C3B-C4B-C12B	-0.42(15)	O3B-C16B-C17B-C18B	179.80(10)
C2B-C3B-C4B-C12B	-174.71(9)	C15B-C16B-C17B-C18B	0.35(17)
C2B-N2B-C5B-C15B	-165.30(10)	C20B-O4B-C18B-C6B	-177.93(13)
C2B-N2B-C5B-C6B	17.62(11)	C20B-O4B-C18B-C17B	1.69(18)
C15B-C5B-C6B-C18B	0.33(16)	C5B-C6B-C18B-O4B	179.52(9)
N2B-C5B-C6B-C18B	177.58(9)	C1B-C6B-C18B-O4B	-4.34(17)
C15B-C5B-C6B-C1B	-176.56(10)	C5B-C6B-C18B-C17B	-0.11(16)
N2B-C5B-C6B-C1B	0.69(12)	C1B-C6B-C18B-C17B	176.03(10)
N1B-C1B-C6B-C18B	56.25(15)	C16B-C17B-C18B-O4B	-179.82(10)
C7B-C1B-C6B-C18B	-71.03(14)	C16B-C17B-C18B-C6B	-0.22(16)

Table S14. Hydrogen bonds and close contacts for (+/-)-**1** [*P*-1] [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N2A-H2A...N2B#1	0.896(15)	2.626(15)	3.4956(13)	164.1(12)
N2A-H2A...O1A	0.896(15)	2.659(15)	3.1569(12)	116.1(11)
N1A-H1A...O4A	0.895(14)	2.527(14)	3.0904(12)	121.5(11)
N1A-H1A...O4B#2	0.895(14)	2.593(14)	3.2205(13)	127.8(11)
N1B-H1B...N1A#2	0.870(17)	2.291(17)	3.1324(13)	162.8(16)
N2B-H2B...O1A#1	0.882(17)	2.590(16)	3.3220(13)	141.0(13)
N2B-H2B...O1B	0.882(17)	2.659(16)	3.1895(13)	119.7(13)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1 #2 -x+1,-y,-z+1

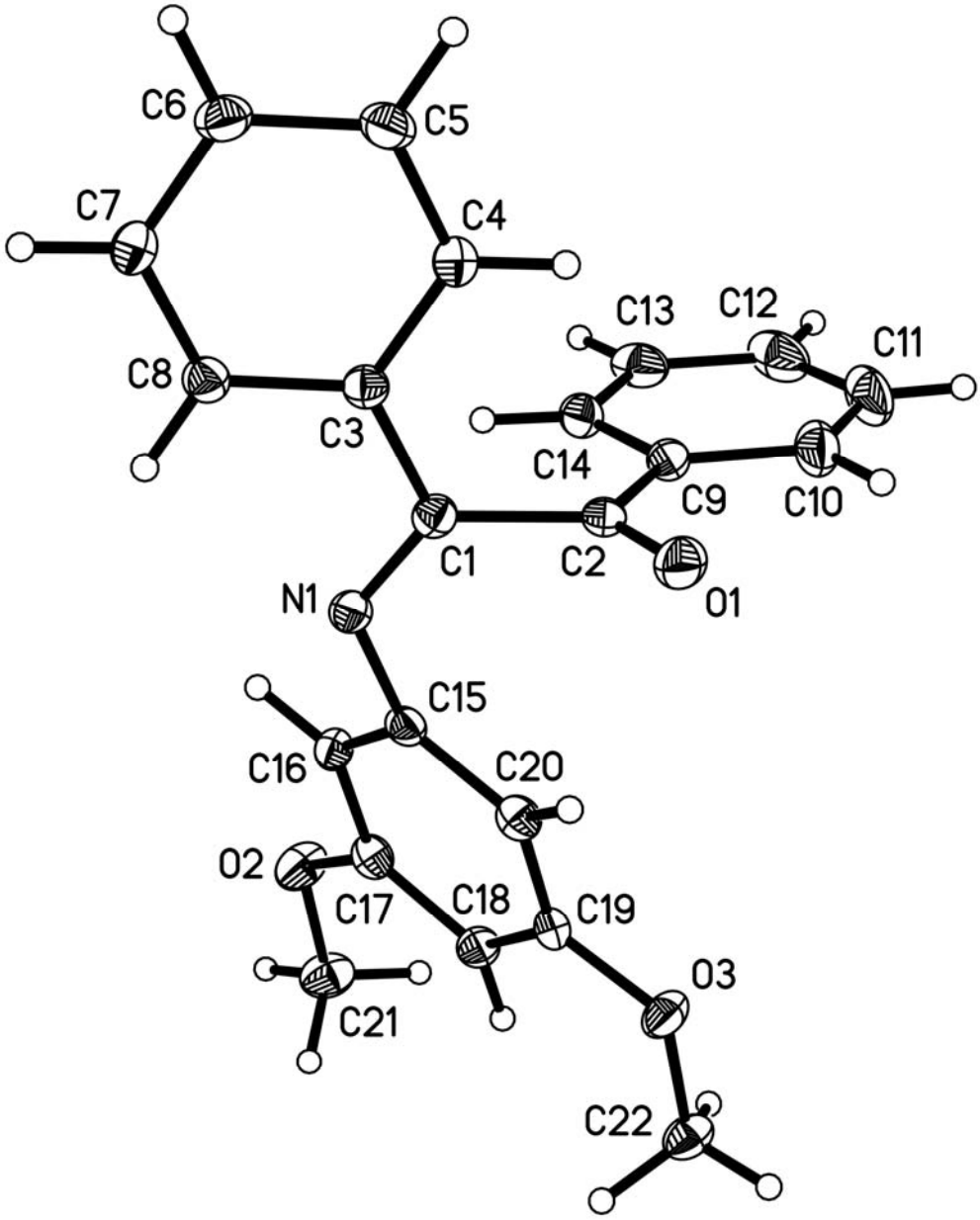


Figure S51. ORTEP diagram of **2** with thermal ellipsoids drawn at 50% probability level.

Experimental Details for the Structure Determination of **2**.

Data collection

A crystal (0.36 x 0.22 x 0.04 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(1) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 3.96 cm. A randomly oriented region of reciprocal space was surveyed: three major sections of frames were collected with 0.50º steps in ω at three different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 3489 strong reflections from the actual data collection after integration.[3] See Table S15 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group $P2_1/c$ was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were found from the difference Fourier map and refined independently from their bonded atoms with individual isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0516$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1126$ (F^2, all data).

Structure description

The structure is the one suggested with all atoms in general positions.

References

[1] *APEX2*, version 2009.3-0; Bruker AXS: Madison, WI, 2009.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.60A; Bruker AXS: Madison, WI, 2008.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma|F_o^2|$$

$$R1 = \Sigma||F_o| - |F_c|| \,/\, \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max(0, F_o^2) + 2/3\, F_c^2$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

Table S15. Crystal data and structure refinement for **2**.

Identification code	**2**, JONJK48	
Empirical formula	C22 H19 N O3	
Formula weight	345.38	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 8.4671(19) Å	α = 90°
	b = 23.820(5) Å	β = 99.090(5)°
	c = 8.7557(18) Å	γ = 90°
Volume	1743.7(6) Å^3	
Z	4	
Density (calculated)	1.316 Mg/m^3	
Absorption coefficient	0.088 mm^{-1}	
F(000)	728	
Crystal color, morphology	colorless, plate	
Crystal size	0.36 x 0.22 x 0.04 mm^3	
Theta range for data collection	2.44 to 29.57°	
Index ranges	$-11 \le h \le 11$, $-29 \le k \le 33$, $-12 \le l \le 11$	
Reflections collected	17805	
Independent reflections	4890 [R(int) = 0.0851]	
Observed reflections	2930	
Completeness to theta = 29.57°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9965 and 0.9691	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4890 / 0 / 311	
Goodness-of-fit on F^2	1.006	
Final R indices [I>2sigma(I)]	R1 = 0.0516, wR2 = 0.0938	
R indices (all data)	R1 = 0.1076, wR2 = 0.1126	
Largest diff. peak and hole	0.289 and -0.249 e.Å^{-3}	

Table S16. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **2**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	518(2)	3482(1)	1455(1)	24(1)
O2	5336(2)	5904(1)	2903(1)	24(1)
O3	2004(2)	5011(1)	-1436(1)	21(1)
N1	1578(2)	4537(1)	3806(2)	16(1)
C1	1274(2)	4010(1)	3738(2)	16(1)
C2	1627(2)	3621(1)	2457(2)	17(1)
C3	563(2)	3740(1)	4993(2)	16(1)
C4	423(2)	3158(1)	5074(2)	18(1)
C5	-143(2)	2905(1)	6307(2)	22(1)
C6	-573(2)	3236(1)	7479(2)	22(1)
C7	-459(2)	3815(1)	7402(2)	20(1)
C8	102(2)	4067(1)	6172(2)	18(1)
C9	3275(2)	3407(1)	2521(2)	19(1)
C10	3612(2)	3015(1)	1436(2)	27(1)
C11	5145(3)	2805(1)	1518(2)	34(1)
C12	6354(3)	2986(1)	2676(2)	32(1)
C13	6032(2)	3371(1)	3759(2)	26(1)
C14	4492(2)	3581(1)	3689(2)	21(1)
C15	2319(2)	4823(1)	2684(2)	16(1)
C16	3488(2)	5212(1)	3253(2)	17(1)
C17	4185(2)	5537(1)	2235(2)	18(1)
C18	3721(2)	5490(1)	644(2)	18(1)
C19	2544(2)	5098(1)	99(2)	17(1)
C20	1834(2)	4765(1)	1098(2)	18(1)
C21	5978(2)	6288(1)	1907(2)	26(1)
C22	2523(2)	5397(1)	-2504(2)	21(1)

Table S17. Bond lengths [Å] and angles [°] for **2**.

O(1)-C(2)	1.2243(19)	C(16)-H(16)	0.973(15)
O(2)-C(17)	1.3706(19)	C(17)-C(18)	1.391(2)
O(2)-C(21)	1.428(2)	C(18)-C(19)	1.393(2)
O(3)-C(19)	1.3651(19)	C(18)-H(18)	0.954(17)
O(3)-C(22)	1.428(2)	C(19)-C(20)	1.386(2)
N(1)-C(1)	1.280(2)	C(20)-H(20)	0.967(18)
N(1)-C(15)	1.420(2)	C(21)-H(21A)	0.996(19)
C(1)-C(3)	1.481(2)	C(21)-H(21B)	1.02(2)
C(1)-C(2)	1.521(2)	C(21)-H(21C)	1.01(2)
C(2)-C(9)	1.478(2)	C(22)-H(22A)	0.946(18)
C(3)-C(4)	1.394(2)	C(22)-H(22B)	1.01(2)
C(3)-C(8)	1.397(2)	C(22)-H(22C)	0.977(19)
C(4)-C(5)	1.387(2)	C(17)-O(2)-C(21)	117.51(13)
C(4)-H(4)	0.969(18)	C(19)-O(3)-C(22)	117.25(13)
C(5)-C(6)	1.387(3)	C(1)-N(1)-C(15)	123.05(14)
C(5)-H(5)	0.991(19)	N(1)-C(1)-C(3)	119.57(15)
C(6)-C(7)	1.385(3)	N(1)-C(1)-C(2)	124.76(15)
C(6)-H(6)	0.957(19)	C(3)-C(1)-C(2)	115.65(14)
C(7)-C(8)	1.381(2)	O(1)-C(2)-C(9)	123.18(15)
C(7)-H(7)	0.956(18)	O(1)-C(2)-C(1)	118.31(15)
C(8)-H(8)	0.99(2)	C(9)-C(2)-C(1)	118.45(14)
C(9)-C(10)	1.394(2)	C(4)-C(3)-C(8)	118.72(16)
C(9)-C(14)	1.395(2)	C(4)-C(3)-C(1)	121.16(15)
C(10)-C(11)	1.382(3)	C(8)-C(3)-C(1)	120.02(15)
C(10)-H(10)	1.004(17)	C(5)-C(4)-C(3)	121.01(17)
C(11)-C(12)	1.390(3)	C(5)-C(4)-H(4)	119.4(11)
C(11)-H(11)	0.92(2)	C(3)-C(4)-H(4)	119.6(11)
C(12)-C(13)	1.377(3)	C(4)-C(5)-C(6)	119.46(18)
C(12)-H(12)	0.94(2)	C(4)-C(5)-H(5)	121.2(10)
C(13)-C(14)	1.389(3)	C(6)-C(5)-H(5)	119.3(10)
C(13)-H(13)	0.99(2)	C(7)-C(6)-C(5)	120.09(17)
C(14)-H(14)	0.949(18)	C(7)-C(6)-H(6)	120.7(11)
C(15)-C(16)	1.391(2)	C(5)-C(6)-H(6)	119.2(11)
C(15)-C(20)	1.392(2)	C(8)-C(7)-C(6)	120.43(17)
C(16)-C(17)	1.380(2)	C(8)-C(7)-H(7)	117.7(11)

C(6)-C(7)-H(7)	121.9(11)	C(17)-C(16)-H(16)	122.4(10)
C(7)-C(8)-C(3)	120.28(17)	C(15)-C(16)-H(16)	118.0(10)
C(7)-C(8)-H(8)	121.1(11)	O(2)-C(17)-C(16)	115.45(14)
C(3)-C(8)-H(8)	118.7(11)	O(2)-C(17)-C(18)	123.26(15)
C(10)-C(9)-C(14)	119.42(17)	C(16)-C(17)-C(18)	121.29(16)
C(10)-C(9)-C(2)	119.91(16)	C(17)-C(18)-C(19)	118.10(16)
C(14)-C(9)-C(2)	120.64(15)	C(17)-C(18)-H(18)	121.4(10)
C(11)-C(10)-C(9)	119.96(18)	C(19)-C(18)-H(18)	120.5(10)
C(11)-C(10)-H(10)	123.5(11)	O(3)-C(19)-C(20)	115.08(15)
C(9)-C(10)-H(10)	116.6(11)	O(3)-C(19)-C(18)	123.26(15)
C(10)-C(11)-C(12)	120.16(19)	C(20)-C(19)-C(18)	121.66(15)
C(10)-C(11)-H(11)	121.2(14)	C(19)-C(20)-C(15)	118.97(16)
C(12)-C(11)-H(11)	118.7(14)	C(19)-C(20)-H(20)	119.8(10)
C(13)-C(12)-C(11)	120.4(2)	C(15)-C(20)-H(20)	121.0(10)
C(13)-C(12)-H(12)	119.4(13)	O(2)-C(21)-H(21A)	107.2(11)
C(11)-C(12)-H(12)	120.2(13)	O(2)-C(21)-H(21B)	111.7(12)
C(12)-C(13)-C(14)	119.73(19)	H(21A)-C(21)-H(21B)	108.9(16)
C(12)-C(13)-H(13)	121.7(12)	O(2)-C(21)-H(21C)	110.0(12)
C(14)-C(13)-H(13)	118.6(12)	H(21A)-C(21)-H(21C)	107.9(15)
C(13)-C(14)-C(9)	120.34(17)	H(21B)-C(21)-H(21C)	111.0(15)
C(13)-C(14)-H(14)	121.7(11)	O(3)-C(22)-H(22A)	105.8(12)
C(9)-C(14)-H(14)	117.9(11)	O(3)-C(22)-H(22B)	110.1(11)
C(16)-C(15)-C(20)	120.29(16)	H(22A)-C(22)-H(22B)	110.7(15)
C(16)-C(15)-N(1)	116.08(14)	O(3)-C(22)-H(22C)	109.1(10)
C(20)-C(15)-N(1)	123.38(15)	H(22A)-C(22)-H(22C)	109.9(15)
C(17)-C(16)-C(15)	119.68(15)	H(22B)-C(22)-H(22C)	111.1(15)

Table S18. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	30(1)	22(1)	20(1)	-1(1)	0(1)	-4(1)
O2	27(1)	26(1)	19(1)	1(1)	2(1)	-11(1)
O3	28(1)	22(1)	13(1)	1(1)	3(1)	-6(1)
N1	17(1)	17(1)	16(1)	1(1)	5(1)	1(1)
C1	15(1)	18(1)	15(1)	1(1)	2(1)	2(1)
C2	23(1)	13(1)	16(1)	2(1)	6(1)	-2(1)
C3	15(1)	16(1)	16(1)	1(1)	2(1)	0(1)
C4	20(1)	18(1)	18(1)	-1(1)	5(1)	2(1)
C5	25(1)	16(1)	24(1)	3(1)	5(1)	1(1)
C6	24(1)	22(1)	20(1)	5(1)	6(1)	0(1)
C7	20(1)	22(1)	18(1)	-1(1)	7(1)	1(1)
C8	18(1)	17(1)	19(1)	1(1)	4(1)	2(1)
C9	26(1)	16(1)	18(1)	1(1)	10(1)	0(1)
C10	36(1)	22(1)	25(1)	-4(1)	13(1)	-2(1)
C11	47(1)	23(1)	39(1)	-4(1)	27(1)	6(1)
C12	31(1)	26(1)	45(1)	8(1)	19(1)	10(1)
C13	26(1)	24(1)	31(1)	8(1)	9(1)	2(1)
C14	24(1)	18(1)	20(1)	1(1)	8(1)	2(1)
C15	18(1)	14(1)	18(1)	2(1)	7(1)	3(1)
C16	19(1)	18(1)	14(1)	-2(1)	2(1)	2(1)
C17	17(1)	16(1)	21(1)	-1(1)	3(1)	-1(1)
C18	20(1)	17(1)	18(1)	2(1)	6(1)	-1(1)
C19	21(1)	14(1)	16(1)	-2(1)	4(1)	4(1)
C20	17(1)	16(1)	20(1)	0(1)	4(1)	0(1)
C21	26(1)	25(1)	25(1)	5(1)	3(1)	-8(1)
C22	24(1)	24(1)	16(1)	4(1)	4(1)	-3(1)

Table S19. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2**.

	x	y	z	$U_{(eq)}$
H4	760(20)	2926(7)	4276(19)	17(5)
H5	-250(20)	2492(8)	6366(19)	23(5)
H6	-940(20)	3059(8)	8340(20)	27(5)
H7	-760(20)	4054(7)	8190(20)	20(5)
H8	190(20)	4482(9)	6110(20)	34(6)
H10	2700(20)	2901(8)	620(20)	23(5)
H11	5390(30)	2545(9)	820(20)	47(7)
H12	7390(30)	2841(9)	2740(20)	42(6)
H13	6870(20)	3499(8)	4600(20)	32(6)
H14	4220(20)	3834(7)	4440(20)	20(5)
H16	3772(19)	5249(7)	4370(18)	10(4)
H18	4200(20)	5715(7)	-61(18)	13(4)
H20	960(20)	4520(7)	691(19)	19(5)
H21A	6720(20)	6542(8)	2580(20)	27(5)
H21B	6600(30)	6085(9)	1160(20)	38(6)
H21C	5090(30)	6525(8)	1330(20)	31(5)
H22A	2020(20)	5281(8)	-3500(20)	25(5)
H22B	2190(20)	5792(9)	-2270(20)	26(5)
H22C	3680(20)	5369(7)	-2436(18)	19(5)

Table S20. Torsion angles [°] for **2**.

C15-N1-C1-C3	177.33(14)	C9-C10-C11-C12	-0.4(3)
C15-N1-C1-C2	-1.0(2)	C10-C11-C12-C13	0.8(3)
N1-C1-C2-O1	-102.2(2)	C11-C12-C13-C14	-0.3(3)
C3-C1-C2-O1	79.36(19)	C12-C13-C14-C9	-0.4(3)
N1-C1-C2-C9	80.6(2)	C10-C9-C14-C13	0.8(3)
C3-C1-C2-C9	-97.83(18)	C2-C9-C14-C13	178.81(16)
N1-C1-C3-C4	-170.83(15)	C1-N1-C15-C16	-137.82(17)
C2-C1-C3-C4	7.7(2)	C1-N1-C15-C20	47.9(2)
N1-C1-C3-C8	5.5(2)	C20-C15-C16-C17	-1.0(3)
C2-C1-C3-C8	-176.01(15)	N1-C15-C16-C17	-175.43(15)
C8-C3-C4-C5	-0.7(3)	C21-O2-C17-C16	-173.86(16)
C1-C3-C4-C5	175.61(16)	C21-O2-C17-C18	5.5(2)
C3-C4-C5-C6	-0.2(3)	C15-C16-C17-O2	-179.34(15)
C4-C5-C6-C7	1.0(3)	C15-C16-C17-C18	1.3(3)
C5-C6-C7-C8	-0.8(3)	O2-C17-C18-C19	179.60(15)
C6-C7-C8-C3	-0.1(3)	C16-C17-C18-C19	-1.1(3)
C4-C3-C8-C7	0.9(3)	C22-O3-C19-C20	171.22(16)
C1-C3-C8-C7	-175.50(15)	C22-O3-C19-C18	-8.8(2)
O1-C2-C9-C10	-2.1(3)	C17-C18-C19-O3	-179.43(16)
C1-C2-C9-C10	174.93(16)	C17-C18-C19-C20	0.6(2)
O1-C2-C9-C14	179.85(16)	O3-C19-C20-C15	179.68(15)
C1-C2-C9-C14	-3.1(2)	C18-C19-C20-C15	-0.3(2)
C14-C9-C10-C11	-0.3(3)	C16-C15-C20-C19	0.5(2)
C2-C9-C10-C11	-178.38(17)	N1-C15-C20-C19	174.55(15)

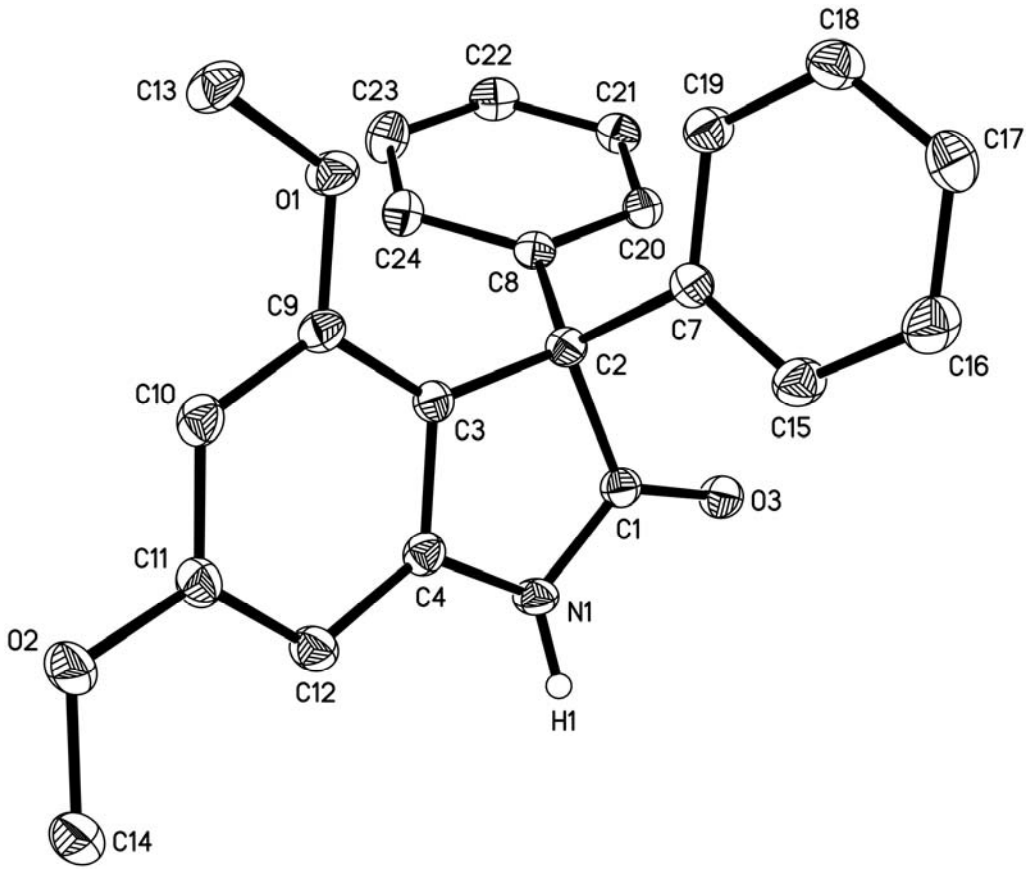


Figure S52. ORTEP diagram of **3** [*P*-1] with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1, to emphasize valency of the nitrogen atoms.

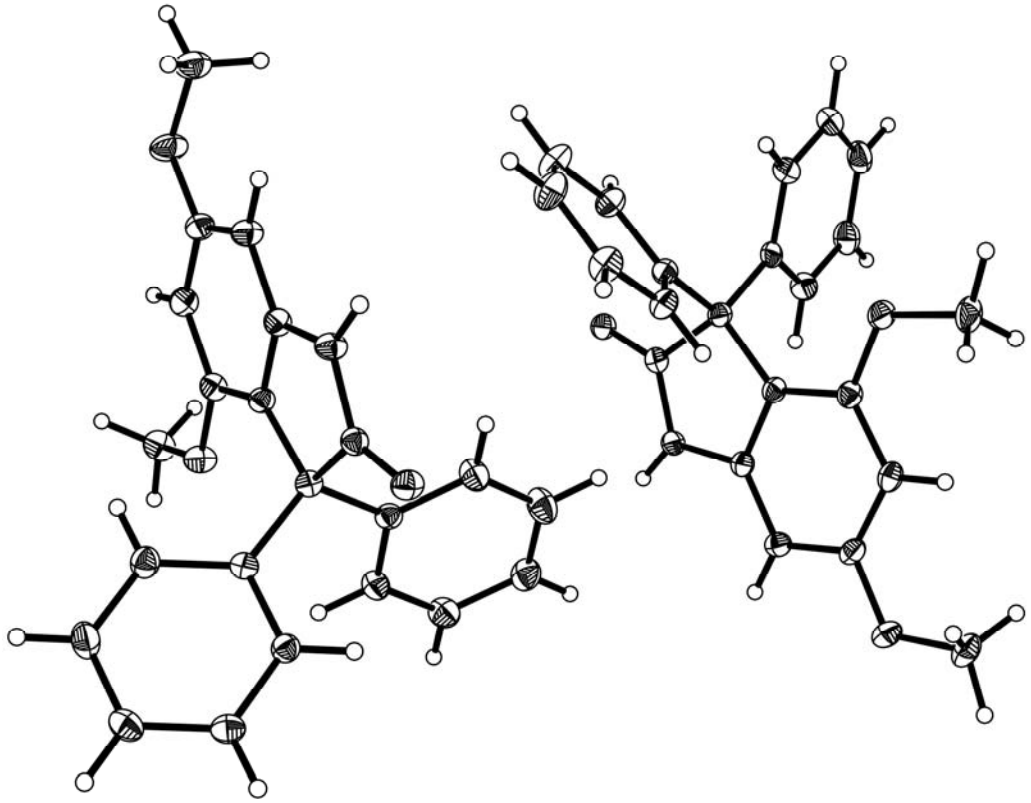


Figure S53. ORTEP diagram of asymmetric unit of **3** [*P*-1] with thermal ellipsoids drawn at 50% probability level.

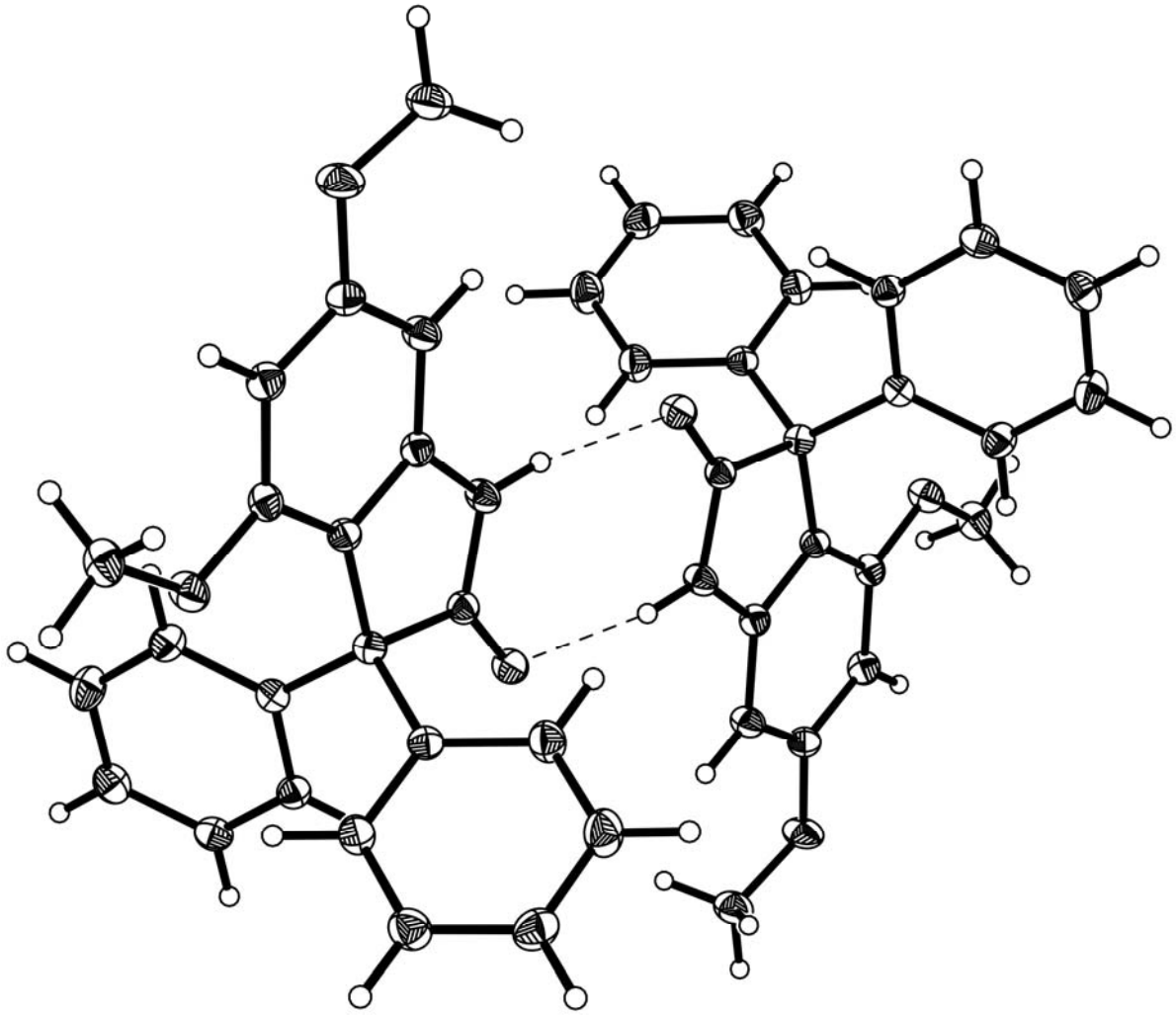


Figure S54. ORTEP diagram of **3** [*P*-1] showing intermolecular hydrogen bonding with thermal ellipsoids drawn at 50% probability level.

Experimental Details for the Structure Determination of **3** [*P*-1].

Data collection

A crystal (0.26 x 0.24 x 0.18 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(1) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 4.00 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4077 strong reflections from the actual data collection after integration.[3] See Table S21 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group *P*-1 was determined based on intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0620$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1631$ (F^2, all data).

Structure description

The structure is the one suggested. There are two independent molecules in the asymmetric unit with all atoms in general positions. Each independent molecule is linked via moderate intermolecular hydrogen bonding to its symmetry equivalent (see Figure S54 and Table S27).

References

[1] *APEX2*, version 2011.4-1; Bruker AXS: Madison, WI, 2011.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.68A; Bruker AXS: Madison, WI, 2009.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma|F_o^2|$$

$$R1 = \Sigma||F_o| - |F_c|| \,/\, \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max(0, F_o^2) + 2/3\, F_c^2$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

Table S21. Crystal data and structure refinement for **3** [*P*-1].

Identification code	**3**, JONJK59	
Empirical formula	C22 H19 N O3	
Formula weight	345.38	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 9.9208(19) Å	α = 78.473(4)°
	b = 13.015(3) Å	β = 82.317(4)°
	c = 13.699(3) Å	γ = 83.248(4)°
Volume	1710.0(6) Å^3	
Z	4	
Density (calculated)	1.342 Mg/m^3	
Absorption coefficient	0.089 mm^{-1}	
F(000)	728	
Crystal color, morphology	colorless, block	
Crystal size	0.26 x 0.24 x 0.18 mm^3	
Theta range for data collection	1.60 to 34.97°	
Index ranges	$-15 \le h \le 15$, $-20 \le k \le 20$, $-22 \le l \le 22$	
Reflections collected	37134	
Independent reflections	14795 [R(int) = 0.0768]	
Observed reflections	8102	
Completeness to theta = 34.97°	98.7%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9841 and 0.9771	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	14795 / 0 / 477	
Goodness-of-fit on F^2	0.992	
Final R indices [I>2sigma(I)]	R1 = 0.0620, wR2 = 0.1337	
R indices (all data)	R1 = 0.1231, wR2 = 0.1631	
Largest diff. peak and hole	0.502 and -0.336 e.Å^{-3}	

Table S22. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3** [*P*-1]. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	623(1)	4370(1)	3699(1)	20(1)
O2	-844(1)	3181(1)	965(1)	25(1)
O3	5150(1)	5592(1)	1096(1)	19(1)
N1	3359(1)	4889(1)	657(1)	17(1)
C1	4033(1)	5238(1)	1302(1)	16(1)
C2	3176(1)	5094(1)	2346(1)	15(1)
C3	1959(1)	4601(1)	2133(1)	15(1)
C4	2146(1)	4478(1)	1140(1)	16(1)
C7	4040(1)	4328(1)	3080(1)	16(1)
C8	2883(1)	6182(1)	2629(1)	14(1)
C9	790(1)	4234(1)	2725(1)	16(1)
C10	-134(2)	3762(1)	2306(1)	18(1)
C11	111(2)	3662(1)	1299(1)	19(1)
C12	1260(2)	4028(1)	689(1)	19(1)
C13	-529(2)	3948(1)	4336(1)	22(1)
C14	-534(2)	2972(1)	-32(1)	25(1)
C15	5003(2)	3576(1)	2740(1)	22(1)
C16	5821(2)	2910(1)	3390(1)	26(1)
C17	5680(2)	2980(1)	4392(1)	24(1)
C18	4700(2)	3707(1)	4744(1)	23(1)
C19	3885(2)	4376(1)	4092(1)	19(1)
C20	3978(2)	6675(1)	2808(1)	16(1)
C21	3776(2)	7670(1)	3047(1)	19(1)
C22	2476(2)	8193(1)	3108(1)	21(1)
C23	1383(2)	7719(1)	2919(1)	24(1)
C24	1583(2)	6714(1)	2684(1)	19(1)
O4	9752(1)	37(1)	1478(1)	21(1)
O5	11178(1)	1528(1)	4120(1)	21(1)
O6	4926(1)	-409(1)	3865(1)	18(1)
N2	6732(1)	299(1)	4296(1)	15(1)
C31	6081(1)	-123(1)	3680(1)	15(1)
C32	7050(1)	-192(1)	2700(1)	15(1)
C33	8300(1)	234(1)	2930(1)	15(1)

C34	8060(1)	522(1)	3867(1)	15(1)
C37	7332(1)	-1358(1)	2622(1)	16(1)
C38	6338(1)	520(1)	1844(1)	16(1)
C39	9571(1)	356(1)	2380(1)	16(1)
C40	10566(1)	795(1)	2760(1)	17(1)
C41	10274(1)	1094(1)	3693(1)	17(1)
C42	9010(2)	953(1)	4273(1)	17(1)
C43	11082(2)	76(1)	928(1)	25(1)
C44	12473(2)	1739(1)	3551(1)	24(1)
C45	7712(2)	-2079(1)	3464(1)	21(1)
C46	7967(2)	-3144(1)	3443(1)	25(1)
C47	7838(2)	-3516(1)	2586(1)	25(1)
C48	7465(2)	-2812(1)	1746(1)	26(1)
C49	7237(2)	-1737(1)	1757(1)	21(1)
C50	5153(2)	239(1)	1562(1)	20(1)
C51	4487(2)	898(1)	810(1)	25(1)
C52	4968(2)	1854(1)	343(1)	26(1)
C53	6121(2)	2144(1)	637(1)	23(1)
C54	6812(2)	1482(1)	1374(1)	19(1)

Table S23. Bond lengths [Å] and angles [°] for **3** [*P*-1].

O(1)-C(9)	1.3663(17)	C(17)-H(17)	0.9500
O(1)-C(13)	1.4346(17)	C(18)-C(19)	1.388(2)
O(2)-C(11)	1.3668(18)	C(18)-H(18)	0.9500
O(2)-C(14)	1.433(2)	C(19)-H(19)	0.9500
O(3)-C(1)	1.2272(17)	C(20)-C(21)	1.384(2)
N(1)-C(1)	1.3504(19)	C(20)-H(20)	0.9500
N(1)-C(4)	1.4029(18)	C(21)-C(22)	1.386(2)
N(1)-H(1)	0.89(2)	C(21)-H(21)	0.9500
C(1)-C(2)	1.552(2)	C(22)-C(23)	1.386(2)
C(2)-C(3)	1.5179(19)	C(22)-H(22)	0.9500
C(2)-C(8)	1.527(2)	C(23)-C(24)	1.395(2)
C(2)-C(7)	1.539(2)	C(23)-H(23)	0.9500
C(3)-C(4)	1.386(2)	C(24)-H(24)	0.9500
C(3)-C(9)	1.3986(19)	O(4)-C(39)	1.3639(17)
C(4)-C(12)	1.382(2)	O(4)-C(43)	1.4300(18)
C(7)-C(19)	1.388(2)	O(5)-C(41)	1.3629(17)
C(7)-C(15)	1.390(2)	O(5)-C(44)	1.4354(18)
C(8)-C(24)	1.390(2)	O(6)-C(31)	1.2256(17)
C(8)-C(20)	1.396(2)	N(2)-C(31)	1.3572(19)
C(9)-C(10)	1.401(2)	N(2)-C(34)	1.4080(18)
C(10)-C(11)	1.398(2)	N(2)-H(2)	0.909(19)
C(10)-H(10)	0.9500	C(31)-C(32)	1.5556(19)
C(11)-C(12)	1.392(2)	C(32)-C(33)	1.5102(19)
C(12)-H(12)	0.9500	C(32)-C(37)	1.534(2)
C(13)-H(13A)	0.9800	C(32)-C(38)	1.537(2)
C(13)-H(13B)	0.9800	C(33)-C(39)	1.3886(19)
C(13)-H(13C)	0.9800	C(33)-C(34)	1.391(2)
C(14)-H(14A)	0.9800	C(34)-C(42)	1.379(2)
C(14)-H(14B)	0.9800	C(37)-C(49)	1.388(2)
C(14)-H(14C)	0.9800	C(37)-C(45)	1.398(2)
C(15)-C(16)	1.387(2)	C(38)-C(54)	1.393(2)
C(15)-H(15)	0.9500	C(38)-C(50)	1.395(2)
C(16)-C(17)	1.381(2)	C(39)-C(40)	1.401(2)
C(16)-H(16)	0.9500	C(40)-C(41)	1.393(2)
C(17)-C(18)	1.384(2)	C(40)-H(40)	0.9500

C(41)-C(42)	1.402(2)	C(3)-C(2)-C(1)	100.98(11)
C(42)-H(42)	0.9500	C(8)-C(2)-C(1)	106.99(11)
C(43)-H(43A)	0.9800	C(7)-C(2)-C(1)	107.13(11)
C(43)-H(43B)	0.9800	C(4)-C(3)-C(9)	117.52(13)
C(43)-H(43C)	0.9800	C(4)-C(3)-C(2)	108.89(12)
C(44)-H(44A)	0.9800	C(9)-C(3)-C(2)	133.54(13)
C(44)-H(44B)	0.9800	C(12)-C(4)-C(3)	125.03(13)
C(44)-H(44C)	0.9800	C(12)-C(4)-N(1)	124.90(13)
C(45)-C(46)	1.385(2)	C(3)-C(4)-N(1)	110.06(12)
C(45)-H(45)	0.9500	C(19)-C(7)-C(15)	118.47(14)
C(46)-C(47)	1.383(2)	C(19)-C(7)-C(2)	120.97(13)
C(46)-H(46)	0.9500	C(15)-C(7)-C(2)	120.56(13)
C(47)-C(48)	1.383(2)	C(24)-C(8)-C(20)	118.83(13)
C(47)-H(47)	0.9500	C(24)-C(8)-C(2)	122.78(13)
C(48)-C(49)	1.393(2)	C(20)-C(8)-C(2)	118.36(12)
C(48)-H(48)	0.9500	O(1)-C(9)-C(3)	116.49(13)
C(49)-H(49)	0.9500	O(1)-C(9)-C(10)	123.81(13)
C(50)-C(51)	1.390(2)	C(3)-C(9)-C(10)	119.70(13)
C(50)-H(50)	0.9500	C(11)-C(10)-C(9)	120.07(13)
C(51)-C(52)	1.387(2)	C(11)-C(10)-H(10)	120.0
C(51)-H(51)	0.9500	C(9)-C(10)-H(10)	120.0
C(52)-C(53)	1.378(2)	O(2)-C(11)-C(12)	122.99(13)
C(52)-H(52)	0.9500	O(2)-C(11)-C(10)	115.53(13)
C(53)-C(54)	1.390(2)	C(12)-C(11)-C(10)	121.48(13)
C(53)-H(53)	0.9500	C(4)-C(12)-C(11)	116.19(14)
C(54)-H(54)	0.9500	C(4)-C(12)-H(12)	121.9
C(9)-O(1)-C(13)	117.48(12)	C(11)-C(12)-H(12)	121.9
C(11)-O(2)-C(14)	116.09(12)	O(1)-C(13)-H(13A)	109.5
C(1)-N(1)-C(4)	111.24(12)	O(1)-C(13)-H(13B)	109.5
C(1)-N(1)-H(1)	122.0(13)	H(13A)-C(13)-H(13B)	109.5
C(4)-N(1)-H(1)	125.3(13)	O(1)-C(13)-H(13C)	109.5
O(3)-C(1)-N(1)	125.36(13)	H(13A)-C(13)-H(13C)	109.5
O(3)-C(1)-C(2)	125.88(13)	H(13B)-C(13)-H(13C)	109.5
N(1)-C(1)-C(2)	108.75(12)	O(2)-C(14)-H(14A)	109.5
C(3)-C(2)-C(8)	116.49(12)	O(2)-C(14)-H(14B)	109.5
C(3)-C(2)-C(7)	112.05(11)	H(14A)-C(14)-H(14B)	109.5
C(8)-C(2)-C(7)	112.03(11)	O(2)-C(14)-H(14C)	109.5

H(14A)-C(14)-H(14C)	109.5	O(6)-C(31)-N(2)	125.91(13)
H(14B)-C(14)-H(14C)	109.5	O(6)-C(31)-C(32)	125.37(13)
C(16)-C(15)-C(7)	120.80(15)	N(2)-C(31)-C(32)	108.72(12)
C(16)-C(15)-H(15)	119.6	C(33)-C(32)-C(37)	111.38(11)
C(7)-C(15)-H(15)	119.6	C(33)-C(32)-C(38)	113.84(12)
C(17)-C(16)-C(15)	120.24(15)	C(37)-C(32)-C(38)	115.64(12)
C(17)-C(16)-H(16)	119.9	C(33)-C(32)-C(31)	100.57(11)
C(15)-C(16)-H(16)	119.9	C(37)-C(32)-C(31)	107.56(11)
C(16)-C(17)-C(18)	119.49(15)	C(38)-C(32)-C(31)	106.38(11)
C(16)-C(17)-H(17)	120.3	C(39)-C(33)-C(34)	118.83(13)
C(18)-C(17)-H(17)	120.3	C(39)-C(33)-C(32)	130.95(13)
C(17)-C(18)-C(19)	120.26(15)	C(34)-C(33)-C(32)	110.21(12)
C(17)-C(18)-H(18)	119.9	C(42)-C(34)-C(33)	123.41(13)
C(19)-C(18)-H(18)	119.9	C(42)-C(34)-N(2)	127.73(13)
C(18)-C(19)-C(7)	120.71(14)	C(33)-C(34)-N(2)	108.86(12)
C(18)-C(19)-H(19)	119.6	C(49)-C(37)-C(45)	118.19(14)
C(7)-C(19)-H(19)	119.6	C(49)-C(37)-C(32)	123.52(13)
C(21)-C(20)-C(8)	120.82(14)	C(45)-C(37)-C(32)	118.29(13)
C(21)-C(20)-H(20)	119.6	C(54)-C(38)-C(50)	118.54(13)
C(8)-C(20)-H(20)	119.6	C(54)-C(38)-C(32)	121.09(13)
C(20)-C(21)-C(22)	120.14(14)	C(50)-C(38)-C(32)	120.27(13)
C(20)-C(21)-H(21)	119.9	O(4)-C(39)-C(33)	116.01(12)
C(22)-C(21)-H(21)	119.9	O(4)-C(39)-C(40)	124.26(13)
C(21)-C(22)-C(23)	119.68(14)	C(33)-C(39)-C(40)	119.72(13)
C(21)-C(22)-H(22)	120.2	C(41)-C(40)-C(39)	119.56(13)
C(23)-C(22)-H(22)	120.2	C(41)-C(40)-H(40)	120.2
C(22)-C(23)-C(24)	120.27(14)	C(39)-C(40)-H(40)	120.2
C(22)-C(23)-H(23)	119.9	O(5)-C(41)-C(40)	123.49(13)
C(24)-C(23)-H(23)	119.9	O(5)-C(41)-C(42)	114.81(13)
C(8)-C(24)-C(23)	120.27(14)	C(40)-C(41)-C(42)	121.69(13)
C(8)-C(24)-H(24)	119.9	C(34)-C(42)-C(41)	116.74(13)
C(23)-C(24)-H(24)	119.9	C(34)-C(42)-H(42)	121.6
C(39)-O(4)-C(43)	117.37(12)	C(41)-C(42)-H(42)	121.6
C(41)-O(5)-C(44)	117.98(12)	O(4)-C(43)-H(43A)	109.5
C(31)-N(2)-C(34)	111.62(12)	O(4)-C(43)-H(43B)	109.5
C(31)-N(2)-H(2)	122.7(12)	H(43A)-C(43)-H(43B)	109.5
C(34)-N(2)-H(2)	125.7(12)	O(4)-C(43)-H(43C)	109.5

H(43A)-C(43)-H(43C)	109.5	C(49)-C(48)-H(48)	119.7
H(43B)-C(43)-H(43C)	109.5	C(37)-C(49)-C(48)	120.64(15)
O(5)-C(44)-H(44A)	109.5	C(37)-C(49)-H(49)	119.7
O(5)-C(44)-H(44B)	109.5	C(48)-C(49)-H(49)	119.7
H(44A)-C(44)-H(44B)	109.5	C(51)-C(50)-C(38)	120.23(14)
O(5)-C(44)-H(44C)	109.5	C(51)-C(50)-H(50)	119.9
H(44A)-C(44)-H(44C)	109.5	C(38)-C(50)-H(50)	119.9
H(44B)-C(44)-H(44C)	109.5	C(52)-C(51)-C(50)	120.81(15)
C(46)-C(45)-C(37)	120.98(15)	C(52)-C(51)-H(51)	119.6
C(46)-C(45)-H(45)	119.5	C(50)-C(51)-H(51)	119.6
C(37)-C(45)-H(45)	119.5	C(53)-C(52)-C(51)	119.06(14)
C(47)-C(46)-C(45)	120.36(15)	C(53)-C(52)-H(52)	120.5
C(47)-C(46)-H(46)	119.8	C(51)-C(52)-H(52)	120.5
C(45)-C(46)-H(46)	119.8	C(52)-C(53)-C(54)	120.68(15)
C(46)-C(47)-C(48)	119.26(15)	C(52)-C(53)-H(53)	119.7
C(46)-C(47)-H(47)	120.4	C(54)-C(53)-H(53)	119.7
C(48)-C(47)-H(47)	120.4	C(53)-C(54)-C(38)	120.65(14)
C(47)-C(48)-C(49)	120.52(15)	C(53)-C(54)-H(54)	119.7
C(47)-C(48)-H(48)	119.7	C(38)-C(54)-H(54)	119.7

Table S24. Anisotropic displacement parameters ($Å^2$ x 10^3) for **3** [*P*-1]. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$h^2 a^{*2} U_{11}$ + ... + 2 h k a* b* U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	20(1)	24(1)	15(1)	-4(1)	3(1)	-8(1)
O2	22(1)	33(1)	24(1)	-11(1)	-1(1)	-13(1)
O3	17(1)	21(1)	18(1)	-4(1)	0(1)	-6(1)
N1	18(1)	21(1)	13(1)	-4(1)	1(1)	-5(1)
C1	16(1)	16(1)	14(1)	-3(1)	-1(1)	-1(1)
C2	15(1)	15(1)	14(1)	-2(1)	-1(1)	-3(1)
C3	14(1)	15(1)	16(1)	-4(1)	-1(1)	-3(1)
C4	14(1)	16(1)	17(1)	-4(1)	0(1)	-3(1)
C7	16(1)	14(1)	17(1)	-3(1)	-2(1)	-3(1)
C8	17(1)	13(1)	13(1)	-1(1)	-1(1)	-3(1)
C9	16(1)	16(1)	15(1)	-3(1)	1(1)	-3(1)
C10	16(1)	19(1)	21(1)	-4(1)	1(1)	-5(1)
C11	17(1)	19(1)	22(1)	-6(1)	-3(1)	-4(1)
C12	19(1)	20(1)	18(1)	-6(1)	-2(1)	-5(1)
C13	21(1)	24(1)	20(1)	-4(1)	6(1)	-7(1)
C14	24(1)	31(1)	24(1)	-10(1)	-3(1)	-8(1)
C15	25(1)	20(1)	19(1)	-4(1)	-2(1)	3(1)
C16	29(1)	20(1)	25(1)	-3(1)	-2(1)	6(1)
C17	24(1)	21(1)	24(1)	0(1)	-8(1)	1(1)
C18	27(1)	22(1)	18(1)	-3(1)	-4(1)	0(1)
C19	20(1)	19(1)	18(1)	-4(1)	-1(1)	0(1)
C20	16(1)	16(1)	17(1)	-3(1)	-2(1)	-2(1)
C21	22(1)	18(1)	18(1)	-3(1)	-2(1)	-5(1)
C22	27(1)	15(1)	22(1)	-4(1)	-2(1)	-2(1)
C23	20(1)	19(1)	30(1)	-4(1)	-2(1)	2(1)
C24	17(1)	19(1)	22(1)	-2(1)	-2(1)	-2(1)
O4	15(1)	34(1)	16(1)	-8(1)	2(1)	-5(1)
O5	18(1)	23(1)	25(1)	-7(1)	-4(1)	-6(1)
O6	15(1)	20(1)	18(1)	-4(1)	1(1)	-4(1)
N2	14(1)	18(1)	13(1)	-4(1)	1(1)	-2(1)
C31	15(1)	13(1)	15(1)	-1(1)	0(1)	-1(1)
C32	14(1)	18(1)	14(1)	-3(1)	0(1)	-3(1)
C33	13(1)	16(1)	15(1)	-2(1)	-1(1)	-2(1)

C34	14(1)	14(1)	15(1)	-1(1)	-2(1)	-1(1)
C37	12(1)	18(1)	18(1)	-5(1)	2(1)	-2(1)
C38	15(1)	18(1)	13(1)	-2(1)	-1(1)	-2(1)
C39	14(1)	18(1)	15(1)	-2(1)	-1(1)	-2(1)
C40	13(1)	18(1)	19(1)	0(1)	-1(1)	-3(1)
C41	15(1)	15(1)	21(1)	-2(1)	-6(1)	-2(1)
C42	18(1)	16(1)	17(1)	-3(1)	-4(1)	-2(1)
C43	14(1)	39(1)	19(1)	-4(1)	3(1)	-2(1)
C44	15(1)	26(1)	30(1)	-4(1)	-3(1)	-6(1)
C45	21(1)	21(1)	20(1)	-3(1)	-2(1)	-1(1)
C46	23(1)	20(1)	30(1)	-1(1)	-1(1)	2(1)
C47	19(1)	19(1)	39(1)	-10(1)	1(1)	0(1)
C48	22(1)	28(1)	30(1)	-15(1)	-2(1)	2(1)
C49	21(1)	24(1)	18(1)	-8(1)	-2(1)	0(1)
C50	18(1)	21(1)	20(1)	0(1)	-3(1)	-5(1)
C51	18(1)	32(1)	25(1)	2(1)	-8(1)	-6(1)
C52	23(1)	27(1)	23(1)	6(1)	-7(1)	-2(1)
C53	23(1)	20(1)	24(1)	2(1)	-4(1)	-4(1)
C54	16(1)	20(1)	21(1)	-2(1)	-3(1)	-3(1)

Table S25. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **3** [*P*-1].

	x	y	z	$U_{(eq)}$
H1	3750(20)	4799(15)	55(15)	31(5)
H10	-928	3510	2707	22
H12	1425	3971	2	22
H13A	-535	4100	5009	33
H13B	-466	3185	4374	33
H13C	-1373	4273	4060	33
H14A	-1283	2633	-198	38
H14B	313	2504	-75	38
H14C	-419	3635	-507	38
H15	5103	3517	2053	26
H16	6481	2404	3146	31
H17	6250	2532	4836	29
H18	4585	3749	5436	27
H19	3214	4872	4342	23
H20	4873	6323	2765	20
H21	4530	7995	3170	23
H22	2336	8873	3280	26
H23	493	8081	2949	28
H24	828	6391	2561	23
H2	6330(20)	420(15)	4905(15)	28(5)
H40	11434	889	2385	21
H42	8818	1144	4915	20
H43A	11080	-174	299	37
H43B	11749	-374	1329	37
H43C	11328	803	779	37
H44A	13017	2052	3941	35
H44B	12325	2229	2923	35
H44C	12960	1079	3400	35
H45	7797	-1835	4060	25
H46	8231	-3622	4021	30
H47	8005	-4248	2573	30
H48	7364	-3062	1157	31
H49	7015	-1259	1168	25

H50	4799	-406	1886	24
H51	3693	692	612	30
H52	4510	2301	-171	31
H53	6446	2805	332	28
H54	7616	1688	1559	23

Table S26. Torsion angles [°] for **3** [*P*-1].

C4-N1-C1-O3	176.59(14)	C2-C3-C9-O1	3.5(2)
C4-N1-C1-C2	-2.19(16)	C4-C3-C9-C10	0.1(2)
O3-C1-C2-C3	-178.13(14)	C2-C3-C9-C10	-176.81(14)
N1-C1-C2-C3	0.65(14)	O1-C9-C10-C11	179.51(14)
O3-C1-C2-C8	59.56(18)	C3-C9-C10-C11	-0.2(2)
N1-C1-C2-C8	-121.66(13)	C14-O2-C11-C12	6.1(2)
O3-C1-C2-C7	-60.74(18)	C14-O2-C11-C10	-173.58(14)
N1-C1-C2-C7	118.04(13)	C9-C10-C11-O2	179.35(13)
C8-C2-C3-C4	116.53(14)	C9-C10-C11-C12	-0.4(2)
C7-C2-C3-C4	-112.62(13)	C3-C4-C12-C11	-1.0(2)
C1-C2-C3-C4	1.11(15)	N1-C4-C12-C11	179.71(14)
C8-C2-C3-C9	-66.3(2)	O2-C11-C12-C4	-178.77(14)
C7-C2-C3-C9	64.5(2)	C10-C11-C12-C4	0.9(2)
C1-C2-C3-C9	178.25(15)	C19-C7-C15-C16	-1.9(2)
C9-C3-C4-C12	0.5(2)	C2-C7-C15-C16	177.51(14)
C2-C3-C4-C12	178.15(13)	C7-C15-C16-C17	0.6(3)
C9-C3-C4-N1	179.86(12)	C15-C16-C17-C18	1.1(3)
C2-C3-C4-N1	-2.48(16)	C16-C17-C18-C19	-1.3(3)
C1-N1-C4-C12	-177.62(14)	C17-C18-C19-C7	-0.1(2)
C1-N1-C4-C3	3.00(17)	C15-C7-C19-C18	1.7(2)
C3-C2-C7-C19	-98.58(16)	C2-C7-C19-C18	-177.74(14)
C8-C2-C7-C19	34.51(18)	C24-C8-C20-C21	0.8(2)
C1-C2-C7-C19	151.54(13)	C2-C8-C20-C21	178.67(13)
C3-C2-C7-C15	81.98(17)	C8-C20-C21-C22	-0.3(2)
C8-C2-C7-C15	-144.93(14)	C20-C21-C22-C23	-0.7(2)
C1-C2-C7-C15	-27.90(18)	C21-C22-C23-C24	1.1(2)
C3-C2-C8-C24	-0.67(19)	C20-C8-C24-C23	-0.3(2)
C7-C2-C8-C24	-131.53(14)	C2-C8-C24-C23	-178.17(14)
C1-C2-C8-C24	111.35(15)	C22-C23-C24-C8	-0.6(2)
C3-C2-C8-C20	-178.50(12)	C34-N2-C31-O6	178.69(13)
C7-C2-C8-C20	50.64(16)	C34-N2-C31-C32	-0.87(16)
C1-C2-C8-C20	-66.48(15)	O6-C31-C32-C33	-178.15(14)
C13-O1-C9-C3	-176.66(13)	N2-C31-C32-C33	1.42(14)
C13-O1-C9-C10	3.6(2)	O6-C31-C32-C37	-61.53(17)
C4-C3-C9-O1	-179.59(13)	N2-C31-C32-C37	118.03(12)

O6-C31-C32-C38	62.94(17)	C32-C33-C39-O4	-0.5(2)
N2-C31-C32-C38	-117.49(12)	C34-C33-C39-C40	-2.1(2)
C37-C32-C33-C39	63.86(19)	C32-C33-C39-C40	178.87(14)
C38-C32-C33-C39	-69.06(19)	O4-C39-C40-C41	-179.92(13)
C31-C32-C33-C39	177.61(15)	C33-C39-C40-C41	0.7(2)
C37-C32-C33-C34	-115.26(13)	C44-O5-C41-C40	3.4(2)
C38-C32-C33-C34	111.82(13)	C44-O5-C41-C42	-177.35(13)
C31-C32-C33-C34	-1.51(14)	C39-C40-C41-O5	-179.89(13)
C39-C33-C34-C42	1.9(2)	C39-C40-C41-C42	0.9(2)
C32-C33-C34-C42	-178.91(13)	C33-C34-C42-C41	-0.2(2)
C39-C33-C34-N2	-178.12(12)	N2-C34-C42-C41	179.75(14)
C32-C33-C34-N2	1.12(16)	O5-C41-C42-C34	179.58(12)
C31-N2-C34-C42	179.89(14)	C40-C41-C42-C34	-1.2(2)
C31-N2-C34-C33	-0.13(16)	C49-C37-C45-C46	-1.0(2)
C33-C32-C37-C49	-118.66(15)	C32-C37-C45-C46	179.35(14)
C38-C32-C37-C49	13.36(19)	C37-C45-C46-C47	-0.5(2)
C31-C32-C37-C49	132.04(14)	C45-C46-C47-C48	0.6(2)
C33-C32-C37-C45	60.93(16)	C46-C47-C48-C49	0.8(2)
C38-C32-C37-C45	-167.05(13)	C45-C37-C49-C48	2.4(2)
C31-C32-C37-C45	-48.37(16)	C32-C37-C49-C48	-177.97(14)
C33-C32-C38-C54	-2.36(19)	C47-C48-C49-C37	-2.4(2)
C37-C32-C38-C54	-133.21(14)	C54-C38-C50-C51	1.6(2)
C31-C32-C38-C54	107.45(15)	C32-C38-C50-C51	178.04(14)
C33-C32-C38-C50	-178.66(13)	C38-C50-C51-C52	-1.5(3)
C37-C32-C38-C50	50.48(18)	C50-C51-C52-C53	0.0(3)
C31-C32-C38-C50	-68.85(16)	C51-C52-C53-C54	1.4(3)
C43-O4-C39-C33	-175.30(13)	C52-C53-C54-C38	-1.2(2)
C43-O4-C39-C40	5.3(2)	C50-C38-C54-C53	-0.3(2)
C34-C33-C39-O4	178.53(12)	C32-C38-C54-C53	-176.67(14)

Table S27. Hydrogen bonds and close contacts for **3** [*P*-1] [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1...O3#1	0.89(2)	1.92(2)	2.7893(17)	168.3(19)
N2-H2...O6#2	0.909(19)	1.96(2)	2.8374(16)	162.4(17)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z #2 -x+1,-y,-z+1

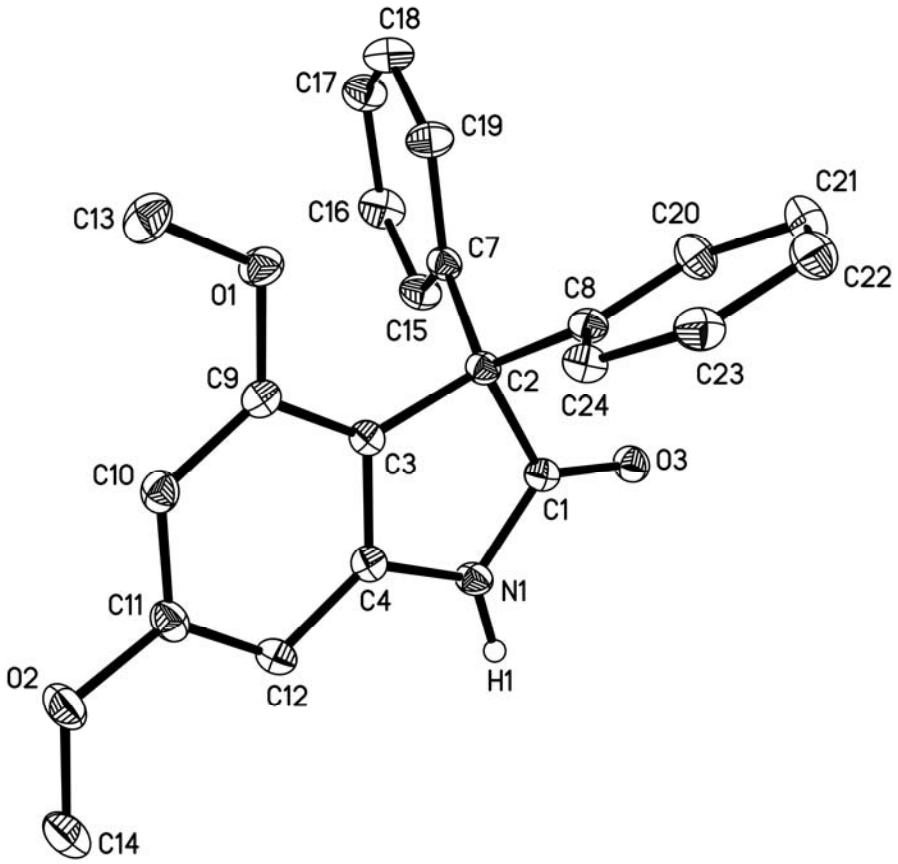


Figure S55. ORTEP diagram of **3** [$P2_1/c$] with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1, to emphasize valency of the nitrogen atoms.

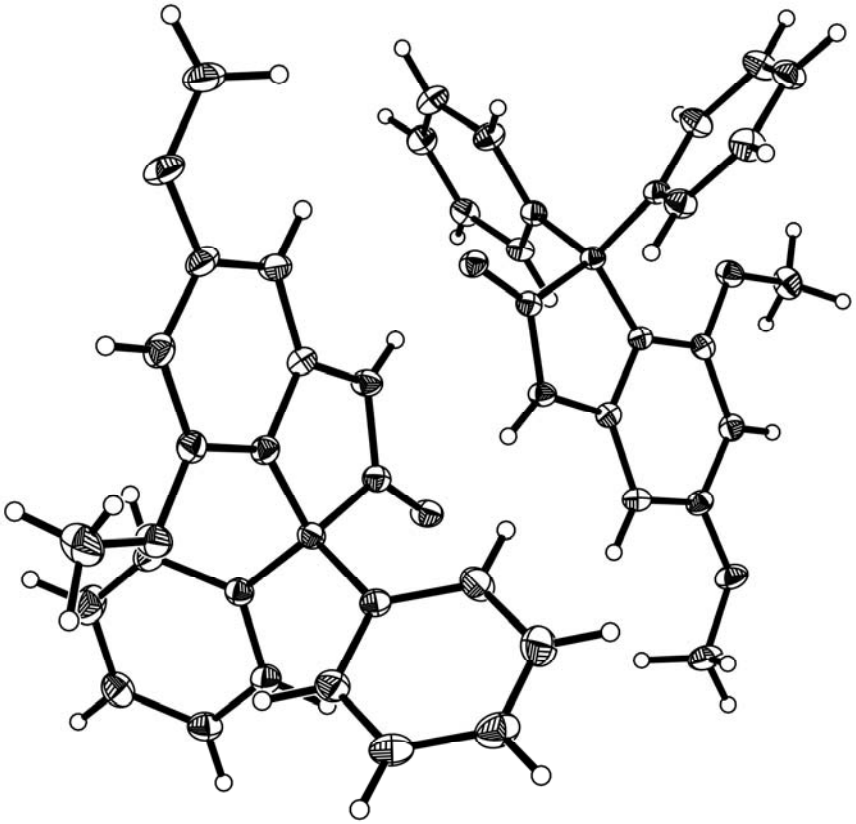


Figure S56. ORTEP diagram of asymmetric unit of **3** [$P2_1/c$] with thermal ellipsoids drawn at 50% probability level.

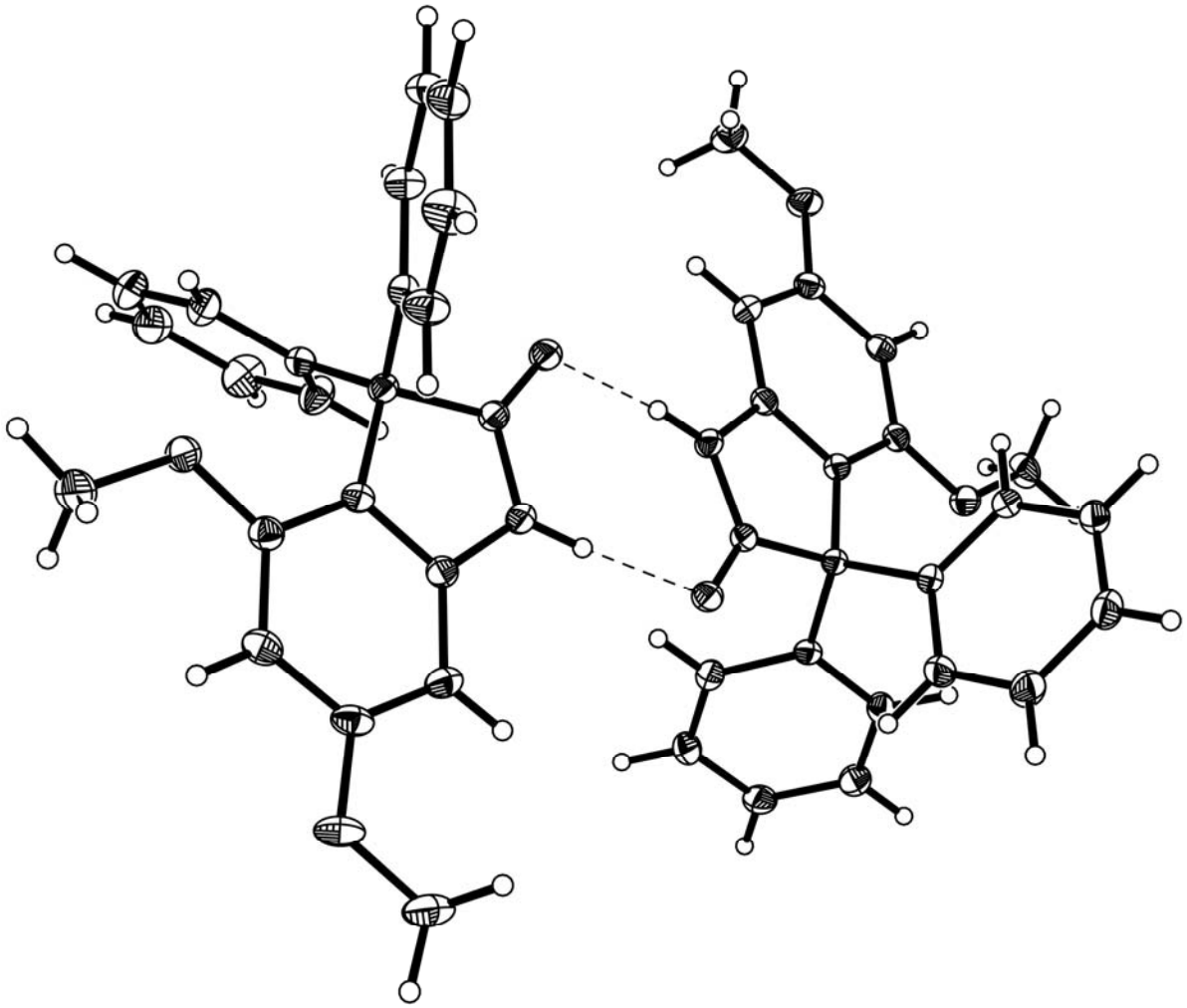


Figure S57. ORTEP diagram of **3** [$P2_1/c$] showing intermolecular hydrogen bonding with thermal ellipsoids drawn at 50% probability level.

Experimental Details for the Structure Determination of **3** [$P2_1/c$].

Data collection

A crystal (0.36 x 0.14 x 0.12 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(1) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 45 seconds and a detector distance of 3.99 cm. A randomly oriented region of reciprocal space was surveyed: five major sections of frames were collected with 0.50º steps in ω at five different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4082 strong reflections from the actual data collection after integration.[3] See Table S28 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group $P2_1/c$ was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. Amine hydrogen atoms H1 and H2 were found from the difference Fourier map, and their positional and isotropic displacement parameters were refined independently from those of all other atoms. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0584$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1503$ (F^2, all data).

Structure description

The structure is the one suggested. There are two independent molecules in the asymmetric unit with all atoms in general positions. Moderate hydrogen bonding links pairs of molecules (see Figure S57 and Table S34).

References

[1] *APEX2*, version 2011.4-1; Bruker AXS: Madison, WI, 2011.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.68A; Bruker AXS: Madison, WI, 2009.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma|F_o^2|$$

$$R1 = \Sigma||F_o| - |F_c|| \,/\, \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max(0, F_o^2) + 2/3\, F_c^2$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

Table S28. Crystal data and structure refinement for **3** [$P2_1/c$].

Identification code	**3**, JONJK60	
Empirical formula	C22 H19 N O3	
Formula weight	345.38	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 13.3622(17) Å	α = 90°
	b = 30.607(4) Å	β = 92.242(3)°
	c = 8.5678(11) Å	γ = 90°
Volume	3501.4(8) Å^3	
Z	8	
Density (calculated)	1.310 Mg/m^3	
Absorption coefficient	0.087 mm^{-1}	
F(000)	1456	
Crystal color, morphology	colorless, rod	
Crystal size	0.36 x 0.14 x 0.12 mm^3	
Theta range for data collection	1.53 to 36.32°	
Index ranges	$-22 \le h \le 22$, $-50 \le k \le 51$, $-14 \le l \le 14$	
Reflections collected	83564	
Independent reflections	16949 [R(int) = 0.0920]	
Observed reflections	9386	
Completeness to theta = 36.32°	99.9%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9896 and 0.9693	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	16949 / 0 / 481	
Goodness-of-fit on F^2	0.931	
Final R indices [I>2sigma(I)]	R1 = 0.0584, wR2 = 0.1282	
R indices (all data)	R1 = 0.1191, wR2 = 0.1503	
Largest diff. peak and hole	0.646 and -0.281 e.Å^{-3}	

Table S29. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **3** [$P2_1/c$]. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	3709(1)	5888(1)	9192(1)	19(1)
O2	5062(1)	4557(1)	7340(1)	22(1)
O3	6847(1)	6722(1)	6438(1)	17(1)
N1	6596(1)	5981(1)	6398(1)	15(1)
C1	6370(1)	6396(1)	6790(1)	14(1)
C2	5403(1)	6392(1)	7748(1)	13(1)
C3	5168(1)	5906(1)	7744(1)	14(1)
C4	5896(1)	5683(1)	6951(1)	14(1)
C7	4588(1)	6660(1)	6880(1)	14(1)
C8	5703(1)	6578(1)	9357(1)	15(1)
C9	4405(1)	5661(1)	8397(1)	15(1)
C10	4395(1)	5209(1)	8217(1)	16(1)
C11	5143(1)	5000(1)	7402(1)	17(1)
C12	5916(1)	5234(1)	6738(1)	17(1)
C13	2945(1)	5641(1)	9920(2)	22(1)
C14	5770(1)	4325(1)	6451(2)	24(1)
C15	4606(1)	6715(1)	5273(1)	19(1)
C16	3840(1)	6933(1)	4452(2)	22(1)
C17	3040(1)	7102(1)	5244(2)	22(1)
C18	3030(1)	7059(1)	6851(2)	23(1)
C19	3797(1)	6842(1)	7668(2)	19(1)
C20	5899(1)	7024(1)	9514(2)	20(1)
C21	6243(1)	7197(1)	10933(2)	22(1)
C22	6395(1)	6924(1)	12220(2)	22(1)
C23	6196(1)	6482(1)	12073(2)	19(1)
C24	5849(1)	6307(1)	10654(1)	16(1)
O4	11190(1)	6601(1)	1336(1)	22(1)
O5	9780(1)	7972(1)	2709(1)	27(1)
O6	8366(1)	5823(1)	4846(1)	19(1)
N2	8514(1)	6564(1)	4541(1)	17(1)
C31	8775(1)	6145(1)	4294(1)	15(1)
C32	9674(1)	6134(1)	3194(1)	14(1)
C33	9817(1)	6620(1)	2932(1)	15(1)

C34	9116(1)	6856(1)	3733(1)	16(1)
C37	10551(1)	5915(1)	4098(1)	16(1)
C38	9372(1)	5897(1)	1670(1)	15(1)
C39	10509(1)	6849(1)	2078(2)	17(1)
C40	10467(1)	7304(1)	2031(2)	20(1)
C41	9739(1)	7528(1)	2843(2)	20(1)
C42	9042(1)	7308(1)	3718(2)	19(1)
C43	11829(1)	6816(1)	298(2)	31(1)
C44	9059(1)	8223(1)	3522(2)	28(1)
C45	11265(1)	6161(1)	4925(2)	23(1)
C46	12047(1)	5958(1)	5765(2)	28(1)
C47	12112(1)	5507(1)	5791(2)	24(1)
C48	11395(1)	5258(1)	4992(2)	22(1)
C49	10620(1)	5462(1)	4150(2)	20(1)
C50	10100(1)	5744(1)	683(2)	21(1)
C51	9836(1)	5550(1)	-736(2)	26(1)
C52	8839(1)	5500(1)	-1195(2)	28(1)
C53	8110(1)	5650(1)	-227(2)	29(1)
C54	8373(1)	5847(1)	1195(2)	23(1)

Table S30. Bond lengths [Å] and angles [°] for **3** [$P2_1/c$].

O(1)-C(9)	1.3637(14)	C(17)-H(17)	0.9500
O(1)-C(13)	1.4333(15)	C(18)-C(19)	1.3886(18)
O(2)-C(11)	1.3606(14)	C(18)-H(18)	0.9500
O(2)-C(14)	1.4259(16)	C(19)-H(19)	0.9500
O(3)-C(1)	1.2274(14)	C(20)-C(21)	1.3884(18)
N(1)-C(1)	1.3506(15)	C(20)-H(20)	0.9500
N(1)-C(4)	1.4007(15)	C(21)-C(22)	1.3911(19)
N(1)-H(1)	0.913(19)	C(21)-H(21)	0.9500
C(1)-C(2)	1.5573(16)	C(22)-C(23)	1.3838(18)
C(2)-C(3)	1.5201(16)	C(22)-H(22)	0.9500
C(2)-C(8)	1.5303(16)	C(23)-C(24)	1.3914(17)
C(2)-C(7)	1.5322(16)	C(23)-H(23)	0.9500
C(3)-C(4)	1.3885(16)	C(24)-H(24)	0.9500
C(3)-C(9)	1.4004(16)	O(4)-C(39)	1.3610(15)
C(4)-C(12)	1.3878(16)	O(4)-C(43)	1.4192(16)
C(7)-C(15)	1.3879(17)	O(5)-C(41)	1.3636(15)
C(7)-C(19)	1.3919(16)	O(5)-C(44)	1.4347(18)
C(8)-C(24)	1.3943(16)	O(6)-C(31)	1.2296(14)
C(8)-C(20)	1.3946(17)	N(2)-C(31)	1.3497(15)
C(9)-C(10)	1.3915(16)	N(2)-C(34)	1.4025(16)
C(10)-C(11)	1.3982(17)	N(2)-H(2)	0.898(17)
C(10)-H(10)	0.9500	C(31)-C(32)	1.5552(16)
C(11)-C(12)	1.3955(17)	C(32)-C(33)	1.5154(16)
C(12)-H(12)	0.9500	C(32)-C(37)	1.5332(17)
C(13)-H(13A)	0.9800	C(32)-C(38)	1.5341(17)
C(13)-H(13B)	0.9800	C(33)-C(34)	1.3856(17)
C(13)-H(13C)	0.9800	C(33)-C(39)	1.3916(17)
C(14)-H(14A)	0.9800	C(34)-C(42)	1.3863(16)
C(14)-H(14B)	0.9800	C(37)-C(45)	1.3880(18)
C(14)-H(14C)	0.9800	C(37)-C(49)	1.3917(17)
C(15)-C(16)	1.3895(18)	C(38)-C(54)	1.3889(18)
C(15)-H(15)	0.9500	C(38)-C(50)	1.3953(17)
C(16)-C(17)	1.3886(19)	C(39)-C(40)	1.3943(17)
C(16)-H(16)	0.9500	C(40)-C(41)	1.3988(18)
C(17)-C(18)	1.3835(19)	C(40)-H(40)	0.9500

C(41)-C(42)	1.3927(18)	C(3)-C(2)-C(1)	100.43(9)
C(42)-H(42)	0.9500	C(8)-C(2)-C(1)	106.16(9)
C(43)-H(43A)	0.9800	C(7)-C(2)-C(1)	109.23(9)
C(43)-H(43B)	0.9800	C(4)-C(3)-C(9)	117.86(10)
C(43)-H(43C)	0.9800	C(4)-C(3)-C(2)	109.52(10)
C(44)-H(44A)	0.9800	C(9)-C(3)-C(2)	132.61(10)
C(44)-H(44B)	0.9800	C(12)-C(4)-C(3)	124.68(11)
C(44)-H(44C)	0.9800	C(12)-C(4)-N(1)	125.78(11)
C(45)-C(46)	1.3914(19)	C(3)-C(4)-N(1)	109.54(10)
C(45)-H(45)	0.9500	C(15)-C(7)-C(19)	118.41(11)
C(46)-C(47)	1.384(2)	C(15)-C(7)-C(2)	120.42(10)
C(46)-H(46)	0.9500	C(19)-C(7)-C(2)	121.13(10)
C(47)-C(48)	1.3846(19)	C(24)-C(8)-C(20)	119.02(11)
C(47)-H(47)	0.9500	C(24)-C(8)-C(2)	121.40(10)
C(48)-C(49)	1.3859(18)	C(20)-C(8)-C(2)	119.45(10)
C(48)-H(48)	0.9500	O(1)-C(9)-C(10)	123.92(11)
C(49)-H(49)	0.9500	O(1)-C(9)-C(3)	116.60(10)
C(50)-C(51)	1.3861(19)	C(10)-C(9)-C(3)	119.47(11)
C(50)-H(50)	0.9500	C(9)-C(10)-C(11)	120.54(11)
C(51)-C(52)	1.383(2)	C(9)-C(10)-H(10)	119.7
C(51)-H(51)	0.9500	C(11)-C(10)-H(10)	119.7
C(52)-C(53)	1.382(2)	O(2)-C(11)-C(12)	123.67(11)
C(52)-H(52)	0.9500	O(2)-C(11)-C(10)	114.83(11)
C(53)-C(54)	1.3924(19)	C(12)-C(11)-C(10)	121.49(11)
C(53)-H(53)	0.9500	C(4)-C(12)-C(11)	115.96(11)
C(54)-H(54)	0.9500	C(4)-C(12)-H(12)	122.0
C(9)-O(1)-C(13)	117.31(10)	C(11)-C(12)-H(12)	122.0
C(11)-O(2)-C(14)	117.54(10)	O(1)-C(13)-H(13A)	109.5
C(1)-N(1)-C(4)	111.69(10)	O(1)-C(13)-H(13B)	109.5
C(1)-N(1)-H(1)	124.1(11)	H(13A)-C(13)-H(13B)	109.5
C(4)-N(1)-H(1)	124.1(11)	O(1)-C(13)-H(13C)	109.5
O(3)-C(1)-N(1)	125.46(11)	H(13A)-C(13)-H(13C)	109.5
O(3)-C(1)-C(2)	125.73(10)	H(13B)-C(13)-H(13C)	109.5
N(1)-C(1)-C(2)	108.81(10)	O(2)-C(14)-H(14A)	109.5
C(3)-C(2)-C(8)	114.33(9)	O(2)-C(14)-H(14B)	109.5
C(3)-C(2)-C(7)	112.36(9)	H(14A)-C(14)-H(14B)	109.5
C(8)-C(2)-C(7)	113.22(9)	O(2)-C(14)-H(14C)	109.5

H(14A)-C(14)-H(14C)	109.5	O(6)-C(31)-N(2)	125.39(11)
H(14B)-C(14)-H(14C)	109.5	O(6)-C(31)-C(32)	125.66(10)
C(7)-C(15)-C(16)	121.28(12)	N(2)-C(31)-C(32)	108.95(10)
C(7)-C(15)-H(15)	119.4	C(33)-C(32)-C(37)	113.92(10)
C(16)-C(15)-H(15)	119.4	C(33)-C(32)-C(38)	111.59(10)
C(17)-C(16)-C(15)	119.85(12)	C(37)-C(32)-C(38)	113.13(9)
C(17)-C(16)-H(16)	120.1	C(33)-C(32)-C(31)	100.06(9)
C(15)-C(16)-H(16)	120.1	C(37)-C(32)-C(31)	107.21(9)
C(18)-C(17)-C(16)	119.22(12)	C(38)-C(32)-C(31)	110.00(9)
C(18)-C(17)-H(17)	120.4	C(34)-C(33)-C(39)	118.27(11)
C(16)-C(17)-H(17)	120.4	C(34)-C(33)-C(32)	110.28(10)
C(17)-C(18)-C(19)	120.78(12)	C(39)-C(33)-C(32)	131.45(11)
C(17)-C(18)-H(18)	119.6	C(33)-C(34)-C(42)	124.41(11)
C(19)-C(18)-H(18)	119.6	C(33)-C(34)-N(2)	108.90(10)
C(18)-C(19)-C(7)	120.42(12)	C(42)-C(34)-N(2)	126.69(11)
C(18)-C(19)-H(19)	119.8	C(45)-C(37)-C(49)	118.71(11)
C(7)-C(19)-H(19)	119.8	C(45)-C(37)-C(32)	121.15(11)
C(21)-C(20)-C(8)	120.80(12)	C(49)-C(37)-C(32)	120.09(11)
C(21)-C(20)-H(20)	119.6	C(54)-C(38)-C(50)	118.03(11)
C(8)-C(20)-H(20)	119.6	C(54)-C(38)-C(32)	121.29(11)
C(20)-C(21)-C(22)	119.89(12)	C(50)-C(38)-C(32)	120.62(11)
C(20)-C(21)-H(21)	120.1	O(4)-C(39)-C(33)	115.82(10)
C(22)-C(21)-H(21)	120.1	O(4)-C(39)-C(40)	124.72(11)
C(23)-C(22)-C(21)	119.56(12)	C(33)-C(39)-C(40)	119.46(11)
C(23)-C(22)-H(22)	120.2	C(39)-C(40)-C(41)	120.27(12)
C(21)-C(22)-H(22)	120.2	C(39)-C(40)-H(40)	119.9
C(22)-C(23)-C(24)	120.77(12)	C(41)-C(40)-H(40)	119.9
C(22)-C(23)-H(23)	119.6	O(5)-C(41)-C(42)	123.90(12)
C(24)-C(23)-H(23)	119.6	O(5)-C(41)-C(40)	114.60(11)
C(23)-C(24)-C(8)	119.96(11)	C(42)-C(41)-C(40)	121.50(11)
C(23)-C(24)-H(24)	120.0	C(34)-C(42)-C(41)	116.09(11)
C(8)-C(24)-H(24)	120.0	C(34)-C(42)-H(42)	122.0
C(39)-O(4)-C(43)	117.59(10)	C(41)-C(42)-H(42)	122.0
C(41)-O(5)-C(44)	117.63(11)	O(4)-C(43)-H(43A)	109.5
C(31)-N(2)-C(34)	111.80(10)	O(4)-C(43)-H(43B)	109.5
C(31)-N(2)-H(2)	122.7(11)	H(43A)-C(43)-H(43B)	109.5
C(34)-N(2)-H(2)	125.4(11)	O(4)-C(43)-H(43C)	109.5

H(43A)-C(43)-H(43C)	109.5	C(49)-C(48)-H(48)	120.1
H(43B)-C(43)-H(43C)	109.5	C(48)-C(49)-C(37)	120.83(12)
O(5)-C(44)-H(44A)	109.5	C(48)-C(49)-H(49)	119.6
O(5)-C(44)-H(44B)	109.5	C(37)-C(49)-H(49)	119.6
H(44A)-C(44)-H(44B)	109.5	C(51)-C(50)-C(38)	121.10(12)
O(5)-C(44)-H(44C)	109.5	C(51)-C(50)-H(50)	119.4
H(44A)-C(44)-H(44C)	109.5	C(38)-C(50)-H(50)	119.4
H(44B)-C(44)-H(44C)	109.5	C(52)-C(51)-C(50)	120.34(13)
C(37)-C(45)-C(46)	120.70(12)	C(52)-C(51)-H(51)	119.8
C(37)-C(45)-H(45)	119.6	C(50)-C(51)-H(51)	119.8
C(46)-C(45)-H(45)	119.6	C(53)-C(52)-C(51)	119.19(13)
C(47)-C(46)-C(45)	119.86(13)	C(53)-C(52)-H(52)	120.4
C(47)-C(46)-H(46)	120.1	C(51)-C(52)-H(52)	120.4
C(45)-C(46)-H(46)	120.1	C(52)-C(53)-C(54)	120.55(13)
C(46)-C(47)-C(48)	120.01(12)	C(52)-C(53)-H(53)	119.7
C(46)-C(47)-H(47)	120.0	C(54)-C(53)-H(53)	119.7
C(48)-C(47)-H(47)	120.0	C(38)-C(54)-C(53)	120.78(12)
C(47)-C(48)-C(49)	119.88(12)	C(38)-C(54)-H(54)	119.6
C(47)-C(48)-H(48)	120.1	C(53)-C(54)-H(54)	119.6

Table S31. Anisotropic displacement parameters ($Å^2$ x 10^3) for **3** [$P2_1/c$]. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$h^2 a^{*2} U_{11}$ + ... + 2 h k a* b* U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	16(1)	19(1)	22(1)	2(1)	6(1)	0(1)
O2	28(1)	12(1)	28(1)	0(1)	4(1)	-2(1)
O3	18(1)	14(1)	18(1)	1(1)	4(1)	-2(1)
N1	16(1)	14(1)	17(1)	0(1)	5(1)	0(1)
C1	15(1)	14(1)	12(1)	0(1)	0(1)	1(1)
C2	14(1)	12(1)	13(1)	0(1)	2(1)	0(1)
C3	15(1)	13(1)	13(1)	0(1)	0(1)	0(1)
C4	16(1)	14(1)	13(1)	1(1)	0(1)	0(1)
C7	16(1)	11(1)	15(1)	1(1)	2(1)	0(1)
C8	15(1)	16(1)	13(1)	0(1)	3(1)	1(1)
C9	14(1)	17(1)	13(1)	2(1)	0(1)	1(1)
C10	17(1)	15(1)	18(1)	3(1)	1(1)	-2(1)
C11	21(1)	13(1)	16(1)	1(1)	-2(1)	0(1)
C12	19(1)	15(1)	17(1)	-1(1)	2(1)	2(1)
C13	18(1)	26(1)	23(1)	5(1)	6(1)	-3(1)
C14	32(1)	15(1)	27(1)	-4(1)	0(1)	2(1)
C15	22(1)	20(1)	16(1)	-1(1)	2(1)	3(1)
C16	29(1)	25(1)	14(1)	1(1)	-1(1)	5(1)
C17	23(1)	20(1)	22(1)	2(1)	-2(1)	6(1)
C18	22(1)	24(1)	22(1)	2(1)	4(1)	8(1)
C19	20(1)	22(1)	16(1)	2(1)	4(1)	5(1)
C20	26(1)	16(1)	17(1)	0(1)	1(1)	-1(1)
C21	29(1)	18(1)	21(1)	-4(1)	2(1)	-4(1)
C22	24(1)	24(1)	16(1)	-4(1)	1(1)	1(1)
C23	21(1)	22(1)	15(1)	0(1)	1(1)	4(1)
C24	18(1)	16(1)	16(1)	0(1)	2(1)	3(1)
O4	21(1)	17(1)	28(1)	4(1)	11(1)	0(1)
O5	33(1)	11(1)	36(1)	0(1)	-1(1)	-1(1)
O6	20(1)	15(1)	22(1)	2(1)	7(1)	1(1)
N2	18(1)	15(1)	17(1)	0(1)	5(1)	1(1)
C31	16(1)	15(1)	15(1)	-1(1)	0(1)	1(1)
C32	14(1)	13(1)	16(1)	0(1)	2(1)	1(1)
C33	16(1)	13(1)	16(1)	0(1)	0(1)	0(1)

C34	18(1)	14(1)	15(1)	-1(1)	0(1)	-1(1)
C37	16(1)	16(1)	15(1)	0(1)	2(1)	0(1)
C38	18(1)	13(1)	15(1)	1(1)	1(1)	1(1)
C39	16(1)	16(1)	19(1)	1(1)	1(1)	-1(1)
C40	20(1)	16(1)	23(1)	3(1)	-1(1)	-3(1)
C41	24(1)	12(1)	22(1)	0(1)	-5(1)	-1(1)
C42	21(1)	14(1)	21(1)	-2(1)	0(1)	2(1)
C43	26(1)	26(1)	43(1)	10(1)	18(1)	0(1)
C44	35(1)	15(1)	33(1)	-3(1)	-8(1)	5(1)
C45	25(1)	17(1)	26(1)	0(1)	-6(1)	0(1)
C46	27(1)	27(1)	30(1)	-1(1)	-11(1)	-3(1)
C47	21(1)	28(1)	22(1)	3(1)	-3(1)	4(1)
C48	24(1)	17(1)	25(1)	1(1)	0(1)	4(1)
C49	19(1)	16(1)	24(1)	0(1)	-2(1)	1(1)
C50	20(1)	21(1)	21(1)	-3(1)	4(1)	1(1)
C51	32(1)	25(1)	21(1)	-5(1)	5(1)	4(1)
C52	38(1)	26(1)	18(1)	-5(1)	-4(1)	4(1)
C53	25(1)	35(1)	26(1)	-6(1)	-8(1)	4(1)
C54	19(1)	29(1)	21(1)	-4(1)	-1(1)	5(1)

Table S32. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **3** [$P2_1/c$].

	x	y	z	$U_{(eq)}$
H1	7159(14)	5902(6)	5900(20)	40(5)
H10	3875	5042	8651	20
H12	6423	5094	6175	20
H13A	2567	5833	10596	33
H13B	3255	5406	10546	33
H13C	2491	5515	9114	33
H14A	5671	4010	6580	37
H14B	6451	4404	6818	37
H14C	5676	4403	5345	37
H15	5153	6602	4724	23
H16	3863	6966	3352	27
H17	2505	7246	4689	26
H18	2493	7180	7402	27
H19	3781	6816	8772	23
H20	5796	7211	8638	23
H21	6375	7501	11026	27
H22	6634	7041	13192	26
H23	6298	6296	12952	23
H24	5712	6003	10569	20
H2	8030(13)	6640(5)	5190(20)	30(4)
H40	10936	7462	1445	24
H42	8545	7458	4271	23
H43A	12200	6599	-287	47
H43B	12302	7002	897	47
H43C	11426	6997	-432	47
H44A	9168	8535	3328	42
H44B	9135	8165	4645	42
H44C	8382	8142	3148	42
H45	11219	6471	4918	27
H46	12536	6130	6320	34
H47	12648	5369	6358	28
H48	11434	4948	5020	26
H49	10131	5289	3601	24

H50	10788	5772	989	25
H51	10344	5450	-1397	31
H52	8657	5366	-2164	33
H53	7423	5618	-534	35
H54	7864	5949	1847	28

Table S33. Torsion angles [°] for **3** [$P2_1/c$].

C4-N1-C1-O3	-178.92(11)	C2-C3-C9-O1	0.83(19)
C4-N1-C1-C2	0.59(13)	C4-C3-C9-C10	-0.74(17)
O3-C1-C2-C3	178.46(11)	C2-C3-C9-C10	-178.84(12)
N1-C1-C2-C3	-1.05(12)	O1-C9-C10-C11	-179.14(11)
O3-C1-C2-C8	-62.24(14)	C3-C9-C10-C11	0.50(18)
N1-C1-C2-C8	118.26(10)	C14-O2-C11-C12	-4.56(18)
O3-C1-C2-C7	60.17(15)	C14-O2-C11-C10	176.54(11)
N1-C1-C2-C7	-119.33(10)	C9-C10-C11-O2	178.49(11)
C8-C2-C3-C4	-112.02(11)	C9-C10-C11-C12	-0.43(19)
C7-C2-C3-C4	117.14(11)	C3-C4-C12-C11	-0.88(18)
C1-C2-C3-C4	1.17(12)	N1-C4-C12-C11	179.60(11)
C8-C2-C3-C9	66.20(16)	O2-C11-C12-C4	-178.25(11)
C7-C2-C3-C9	-64.64(16)	C10-C11-C12-C4	0.58(18)
C1-C2-C3-C9	179.39(12)	C19-C7-C15-C16	-2.02(19)
C9-C3-C4-C12	0.97(18)	C2-C7-C15-C16	175.74(12)
C2-C3-C4-C12	179.49(11)	C7-C15-C16-C17	0.4(2)
C9-C3-C4-N1	-179.44(10)	C15-C16-C17-C18	1.4(2)
C2-C3-C4-N1	-0.92(13)	C16-C17-C18-C19	-1.5(2)
C1-N1-C4-C12	179.78(12)	C17-C18-C19-C7	-0.2(2)
C1-N1-C4-C3	0.20(14)	C15-C7-C19-C18	1.96(19)
C3-C2-C7-C15	-85.31(13)	C2-C7-C19-C18	-175.78(12)
C8-C2-C7-C15	143.28(11)	C24-C8-C20-C21	0.58(19)
C1-C2-C7-C15	25.22(15)	C2-C8-C20-C21	-175.30(12)
C3-C2-C7-C19	92.38(13)	C8-C20-C21-C22	-0.1(2)
C8-C2-C7-C19	-39.02(15)	C20-C21-C22-C23	-0.3(2)
C1-C2-C7-C19	-157.08(11)	C21-C22-C23-C24	0.18(19)
C3-C2-C8-C24	6.65(15)	C22-C23-C24-C8	0.35(19)
C7-C2-C8-C24	137.07(11)	C20-C8-C24-C23	-0.72(18)
C1-C2-C8-C24	-103.10(12)	C2-C8-C24-C23	175.07(11)
C3-C2-C8-C20	-177.58(11)	C34-N2-C31-O6	-179.21(12)
C7-C2-C8-C20	-47.16(14)	C34-N2-C31-C32	0.58(14)
C1-C2-C8-C20	72.68(13)	O6-C31-C32-C33	179.72(12)
C13-O1-C9-C10	2.04(17)	N2-C31-C32-C33	-0.07(12)
C13-O1-C9-C3	-177.61(11)	O6-C31-C32-C37	-61.20(15)
C4-C3-C9-O1	178.93(10)	N2-C31-C32-C37	119.00(11)

O6-C31-C32-C38	62.19(15)	C32-C33-C39-O4	-0.2(2)
N2-C31-C32-C38	-117.61(11)	C34-C33-C39-C40	-1.06(18)
C37-C32-C33-C34	-114.50(11)	C32-C33-C39-C40	179.80(12)
C38-C32-C33-C34	115.88(11)	O4-C39-C40-C41	-179.65(12)
C31-C32-C33-C34	-0.46(13)	C33-C39-C40-C41	0.31(19)
C37-C32-C33-C39	64.69(17)	C44-O5-C41-C42	-0.01(18)
C38-C32-C33-C39	-64.92(17)	C44-O5-C41-C40	-179.55(11)
C31-C32-C33-C39	178.73(13)	C39-C40-C41-O5	179.76(11)
C39-C33-C34-C42	1.39(19)	C39-C40-C41-C42	0.21(19)
C32-C33-C34-C42	-179.30(11)	C33-C34-C42-C41	-0.87(19)
C39-C33-C34-N2	-178.49(11)	N2-C34-C42-C41	178.98(12)
C32-C33-C34-N2	0.83(14)	O5-C41-C42-C34	-179.46(12)
C31-N2-C34-C33	-0.90(14)	C40-C41-C42-C34	0.05(18)
C31-N2-C34-C42	179.23(12)	C49-C37-C45-C46	1.3(2)
C33-C32-C37-C45	15.63(16)	C32-C37-C45-C46	178.78(13)
C38-C32-C37-C45	144.47(12)	C37-C45-C46-C47	-0.6(2)
C31-C32-C37-C45	-94.09(13)	C45-C46-C47-C48	-0.4(2)
C33-C32-C37-C49	-166.90(11)	C46-C47-C48-C49	0.8(2)
C38-C32-C37-C49	-38.06(15)	C47-C48-C49-C37	-0.1(2)
C31-C32-C37-C49	83.38(13)	C45-C37-C49-C48	-0.92(19)
C33-C32-C38-C54	-89.92(14)	C32-C37-C49-C48	-178.45(12)
C37-C32-C38-C54	140.05(12)	C54-C38-C50-C51	0.52(19)
C31-C32-C38-C54	20.19(15)	C32-C38-C50-C51	-176.53(12)
C33-C32-C38-C50	87.03(13)	C38-C50-C51-C52	-0.7(2)
C37-C32-C38-C50	-43.00(15)	C50-C51-C52-C53	0.5(2)
C31-C32-C38-C50	-162.86(11)	C51-C52-C53-C54	-0.1(2)
C43-O4-C39-C33	172.36(12)	C50-C38-C54-C53	-0.1(2)
C43-O4-C39-C40	-7.69(19)	C32-C38-C54-C53	176.90(12)
C34-C33-C39-O4	178.90(11)	C52-C53-C54-C38	-0.1(2)

Table S34. Hydrogen bonds and close contacts for **3** [$P2_1/c$] [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1...O6	0.913(19)	1.893(19)	2.7999(13)	172.1(17)
N2-H2...O3	0.898(17)	1.960(17)	2.8498(14)	170.2(16)

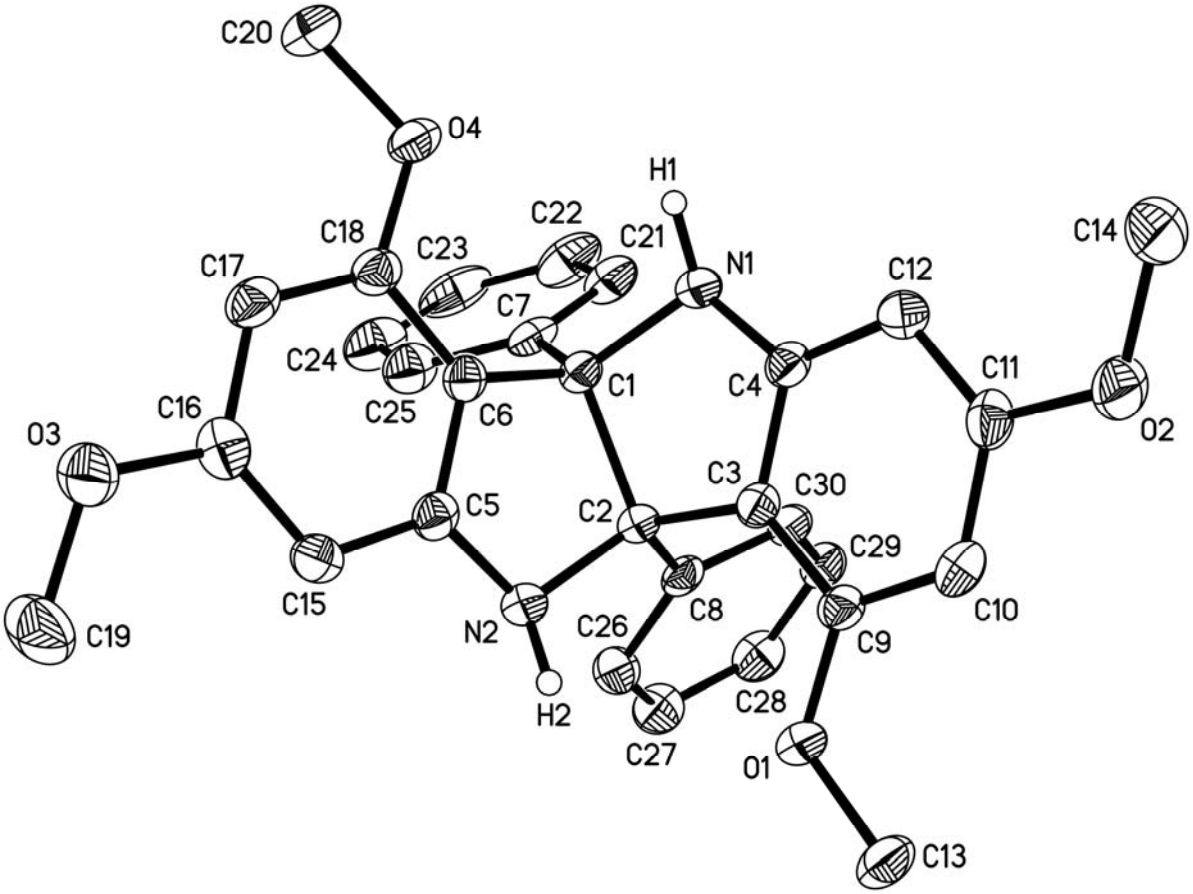


Figure S58. ORTEP diagram of **4** with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1 and H2, to emphasize valency of the nitrogen atoms.

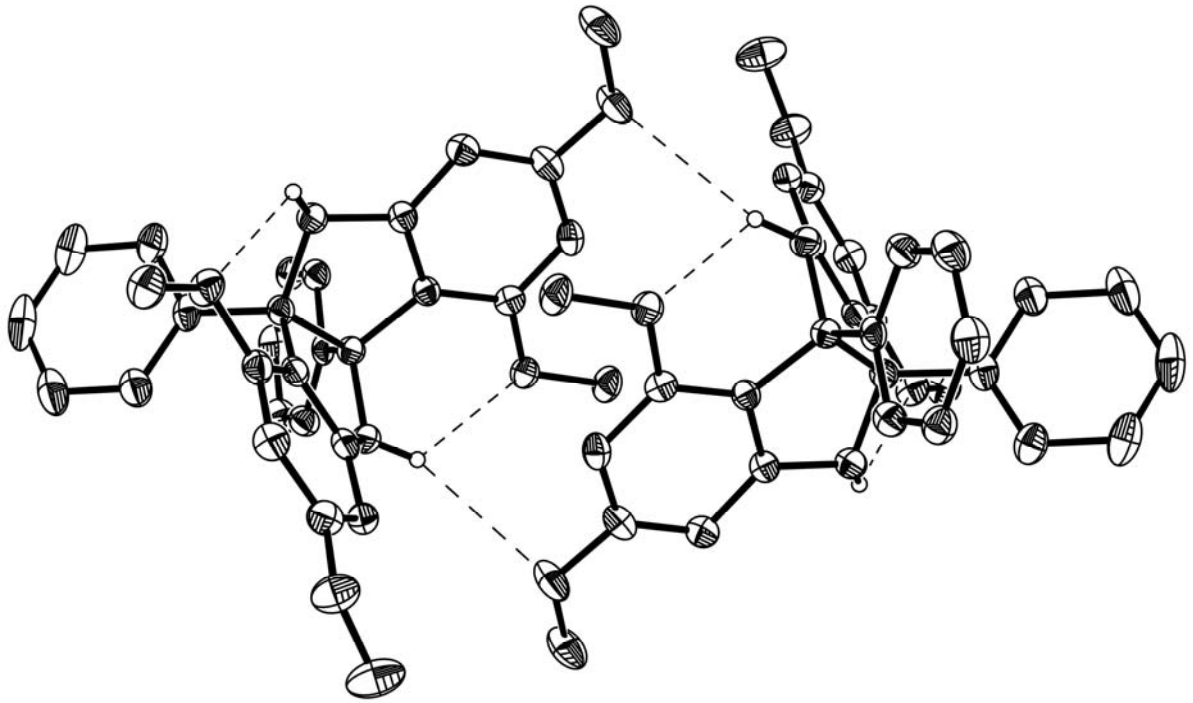


Figure S59. ORTEP diagram of (+/-)-**4** with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1, H2, H1A, and H2A, to emphasize intermolecular and intramolecular hydrogen bonding.

Experimental Details for the Structure Determination of (+/-)-**4**.

Data collection

A crystal (0.20 x 0.14 x 0.10 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(1) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 4.01 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4064 strong reflections from the actual data collection after integration.[3] See Table S35 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXS-97[4] and refined using SHELXL-97.[4] The space group $P2_1/c$ was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. Amine hydrogen atoms were found from the difference Fourier map and their positional and isotropic displacement parameters were refined independently from those of all other atoms. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0515$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1210$ (F^2, all data).

Structure description

The structure is the one suggested with all atoms in general positions. Weak intermolecular and intramolecular hydrogen bonding is observed (see Figure S59 and Table S41).

References

[1] *APEX2*, version 2010.3-0; Bruker AXS: Madison, WI, 2010.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.68A; Bruker AXS: Madison, WI, 2009.

[4] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \; / \; \Sigma|F_o^2|$$

$$R1 = \Sigma||F_o| - |F_c|| \; / \; \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \; / \; \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = 1 \; / \; [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max(0, F_o^2) + 2/3 \, F_c^2$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \; / \; (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

Table S35. Crystal data and structure refinement for (+/-)-**4**.

Identification code	(+/-)-**4**, JONJK56	
Empirical formula	C30 H28 N2 O4	
Formula weight	480.54	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 10.8867(6) Å	α = 90°
	b = 11.7612(7) Å	β = 100.1780(10)°
	c = 19.4486(11) Å	γ = 90°
Volume	2451.0(2) Å^3	
Z	4	
Density (calculated)	1.302 Mg/m^3	
Absorption coefficient	0.087 mm^{-1}	
F(000)	1016	
Crystal color, morphology	colorless, plate	
Crystal size	0.20 x 0.14 x 0.10 mm^3	
Theta range for data collection	1.90 to 28.28°	
Index ranges	$-14 \le h \le 14$, $-15 \le k \le 15$, $-25 \le l \le 25$	
Reflections collected	38229	
Independent reflections	6096 [R(int) = 0.1120]	
Observed reflections	3542	
Completeness to theta = 28.28°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9914 and 0.9829	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6096 / 0 / 337	
Goodness-of-fit on F^2	1.005	
Final R indices [I>2sigma(I)]	R1 = 0.0515, wR2 = 0.0969	
R indices (all data)	R1 = 0.1086, wR2 = 0.1210	
Largest diff. peak and hole	0.289 and -0.257 e.Å^{-3}	

Table S36. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for (+/-)-**4**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	1184(1)	1448(1)	5630(1)	24(1)
O2	-3115(1)	1272(1)	4606(1)	35(1)
O3	3522(1)	-1792(1)	3004(1)	35(1)
O4	1030(1)	1400(1)	2104(1)	26(1)
N1	132(2)	2681(2)	3354(1)	23(1)
N2	2715(2)	1417(2)	4459(1)	22(1)
C1	1493(2)	2469(2)	3496(1)	20(1)
C2	1803(2)	2355(2)	4337(1)	20(1)
C3	533(2)	2029(2)	4479(1)	20(1)
C4	-382(2)	2211(2)	3898(1)	20(1)
C5	2563(2)	743(2)	3856(1)	22(1)
C6	1875(2)	1298(2)	3291(1)	20(1)
C7	2205(2)	3410(2)	3204(1)	24(1)
C8	2299(2)	3428(2)	4724(1)	21(1)
C9	211(2)	1605(2)	5088(1)	21(1)
C10	-1025(2)	1364(2)	5112(1)	24(1)
C11	-1925(2)	1551(2)	4518(1)	25(1)
C12	-1640(2)	1977(2)	3901(1)	24(1)
C13	897(2)	994(2)	6268(1)	30(1)
C14	-4085(2)	1348(2)	4015(1)	39(1)
C15	3127(2)	-304(2)	3801(1)	24(1)
C16	2985(2)	-772(2)	3137(1)	26(1)
C17	2296(2)	-241(2)	2552(1)	26(1)
C18	1734(2)	800(2)	2634(1)	23(1)
C19	4402(2)	-2257(2)	3565(1)	46(1)
C20	1079(2)	1036(2)	1408(1)	30(1)
C21	1706(2)	4494(2)	3081(1)	31(1)
C22	2398(3)	5358(2)	2854(1)	40(1)
C23	3583(3)	5150(2)	2736(1)	43(1)
C24	4089(2)	4083(2)	2846(1)	40(1)
C25	3403(2)	3215(2)	3079(1)	31(1)
C26	3527(2)	3519(2)	5068(1)	26(1)
C27	3949(2)	4515(2)	5420(1)	31(1)

C28	3161(2)	5436(2)	5413(1)	32(1)
C29	1941(2)	5362(2)	5061(1)	27(1)
C30	1512(2)	4360(2)	4732(1)	24(1)

Table S37. Bond lengths [Å] and angles [°] for (+/-)-**4**.

O(1)-C(9)	1.368(2)	C(13)-H(13B)	0.9800
O(1)-C(13)	1.434(2)	C(13)-H(13C)	0.9800
O(2)-C(11)	1.376(2)	C(14)-H(14A)	0.9800
O(2)-C(14)	1.421(2)	C(14)-H(14B)	0.9800
O(3)-C(16)	1.378(2)	C(14)-H(14C)	0.9800
O(3)-C(19)	1.428(3)	C(15)-C(16)	1.386(3)
O(4)-C(18)	1.367(2)	C(15)-H(15A)	0.9500
O(4)-C(20)	1.431(2)	C(16)-C(17)	1.396(3)
N(1)-C(4)	1.396(2)	C(17)-C(18)	1.390(3)
N(1)-C(1)	1.480(2)	C(17)-H(17A)	0.9500
N(1)-H(1)	0.91(2)	C(19)-H(19A)	0.9800
N(2)-C(5)	1.401(2)	C(19)-H(19B)	0.9800
N(2)-C(2)	1.476(2)	C(19)-H(19C)	0.9800
N(2)-H(2)	0.89(2)	C(20)-H(20A)	0.9800
C(1)-C(6)	1.512(3)	C(20)-H(20B)	0.9800
C(1)-C(7)	1.518(3)	C(20)-H(20C)	0.9800
C(1)-C(2)	1.616(2)	C(21)-C(22)	1.384(3)
C(2)-C(3)	1.506(3)	C(21)-H(21A)	0.9500
C(2)-C(8)	1.519(3)	C(22)-C(23)	1.371(4)
C(3)-C(4)	1.384(3)	C(22)-H(22A)	0.9500
C(3)-C(9)	1.387(3)	C(23)-C(24)	1.373(4)
C(4)-C(12)	1.397(3)	C(23)-H(23A)	0.9500
C(5)-C(6)	1.379(3)	C(24)-C(25)	1.387(3)
C(5)-C(15)	1.389(3)	C(24)-H(24A)	0.9500
C(6)-C(18)	1.390(3)	C(25)-H(25A)	0.9500
C(7)-C(25)	1.387(3)	C(26)-C(27)	1.393(3)
C(7)-C(21)	1.389(3)	C(26)-H(26A)	0.9500
C(8)-C(26)	1.389(3)	C(27)-C(28)	1.381(3)
C(8)-C(30)	1.393(3)	C(27)-H(27A)	0.9500
C(9)-C(10)	1.385(3)	C(28)-C(29)	1.386(3)
C(10)-C(11)	1.394(3)	C(28)-H(28A)	0.9500
C(10)-H(10A)	0.9500	C(29)-C(30)	1.383(3)
C(11)-C(12)	1.385(3)	C(29)-H(29A)	0.9500
C(12)-H(12A)	0.9500	C(30)-H(30A)	0.9500
C(13)-H(13A)	0.9800	C(9)-O(1)-C(13)	117.33(15)

C(11)-O(2)-C(14)	117.72(16)	C(26)-C(8)-C(2)	122.02(18)
C(16)-O(3)-C(19)	116.05(17)	C(30)-C(8)-C(2)	119.55(17)
C(18)-O(4)-C(20)	116.67(16)	O(1)-C(9)-C(10)	124.83(17)
C(4)-N(1)-C(1)	108.33(15)	O(1)-C(9)-C(3)	115.42(17)
C(4)-N(1)-H(1)	116.6(13)	C(10)-C(9)-C(3)	119.74(17)
C(1)-N(1)-H(1)	112.3(13)	C(9)-C(10)-C(11)	119.01(18)
C(5)-N(2)-C(2)	108.13(15)	C(9)-C(10)-H(10A)	120.5
C(5)-N(2)-H(2)	115.4(15)	C(11)-C(10)-H(10A)	120.5
C(2)-N(2)-H(2)	114.1(15)	O(2)-C(11)-C(12)	123.69(18)
N(1)-C(1)-C(6)	114.67(16)	O(2)-C(11)-C(10)	113.44(18)
N(1)-C(1)-C(7)	111.59(16)	C(12)-C(11)-C(10)	122.87(18)
C(6)-C(1)-C(7)	112.54(16)	C(11)-C(12)-C(4)	116.41(18)
N(1)-C(1)-C(2)	103.03(14)	C(11)-C(12)-H(12A)	121.8
C(6)-C(1)-C(2)	99.94(15)	C(4)-C(12)-H(12A)	121.8
C(7)-C(1)-C(2)	114.24(15)	O(1)-C(13)-H(13A)	109.5
N(2)-C(2)-C(3)	113.19(16)	O(1)-C(13)-H(13B)	109.5
N(2)-C(2)-C(8)	111.78(15)	H(13A)-C(13)-H(13B)	109.5
C(3)-C(2)-C(8)	112.24(16)	O(1)-C(13)-H(13C)	109.5
N(2)-C(2)-C(1)	103.84(15)	H(13A)-C(13)-H(13C)	109.5
C(3)-C(2)-C(1)	99.93(14)	H(13B)-C(13)-H(13C)	109.5
C(8)-C(2)-C(1)	115.11(16)	O(2)-C(14)-H(14A)	109.5
C(4)-C(3)-C(9)	119.93(17)	O(2)-C(14)-H(14B)	109.5
C(4)-C(3)-C(2)	111.28(17)	H(14A)-C(14)-H(14B)	109.5
C(9)-C(3)-C(2)	128.78(17)	O(2)-C(14)-H(14C)	109.5
C(3)-C(4)-N(1)	110.63(17)	H(14A)-C(14)-H(14C)	109.5
C(3)-C(4)-C(12)	122.04(18)	H(14B)-C(14)-H(14C)	109.5
N(1)-C(4)-C(12)	127.29(17)	C(16)-C(15)-C(5)	116.55(19)
C(6)-C(5)-C(15)	122.88(19)	C(16)-C(15)-H(15A)	121.7
C(6)-C(5)-N(2)	111.51(18)	C(5)-C(15)-H(15A)	121.7
C(15)-C(5)-N(2)	125.31(18)	O(3)-C(16)-C(15)	122.69(19)
C(5)-C(6)-C(18)	119.30(18)	O(3)-C(16)-C(17)	114.89(18)
C(5)-C(6)-C(1)	110.93(17)	C(15)-C(16)-C(17)	122.41(19)
C(18)-C(6)-C(1)	129.42(18)	C(18)-C(17)-C(16)	119.04(19)
C(25)-C(7)-C(21)	118.3(2)	C(18)-C(17)-H(17A)	120.5
C(25)-C(7)-C(1)	120.17(19)	C(16)-C(17)-H(17A)	120.5
C(21)-C(7)-C(1)	121.50(18)	O(4)-C(18)-C(17)	124.63(18)
C(26)-C(8)-C(30)	118.43(19)	O(4)-C(18)-C(6)	115.56(18)

C(17)-C(18)-C(6)	119.81(18)	C(23)-C(24)-C(25)	119.9(2)
O(3)-C(19)-H(19A)	109.5	C(23)-C(24)-H(24A)	120.0
O(3)-C(19)-H(19B)	109.5	C(25)-C(24)-H(24A)	120.0
H(19A)-C(19)-H(19B)	109.5	C(7)-C(25)-C(24)	120.8(2)
O(3)-C(19)-H(19C)	109.5	C(7)-C(25)-H(25A)	119.6
H(19A)-C(19)-H(19C)	109.5	C(24)-C(25)-H(25A)	119.6
H(19B)-C(19)-H(19C)	109.5	C(8)-C(26)-C(27)	120.5(2)
O(4)-C(20)-H(20A)	109.5	C(8)-C(26)-H(26A)	119.8
O(4)-C(20)-H(20B)	109.5	C(27)-C(26)-H(26A)	119.8
H(20A)-C(20)-H(20B)	109.5	C(28)-C(27)-C(26)	120.3(2)
O(4)-C(20)-H(20C)	109.5	C(28)-C(27)-H(27A)	119.8
H(20A)-C(20)-H(20C)	109.5	C(26)-C(27)-H(27A)	119.8
H(20B)-C(20)-H(20C)	109.5	C(27)-C(28)-C(29)	119.7(2)
C(22)-C(21)-C(7)	120.6(2)	C(27)-C(28)-H(28A)	120.1
C(22)-C(21)-H(21A)	119.7	C(29)-C(28)-H(28A)	120.1
C(7)-C(21)-H(21A)	119.7	C(30)-C(29)-C(28)	119.9(2)
C(23)-C(22)-C(21)	120.3(2)	C(30)-C(29)-H(29A)	120.1
C(23)-C(22)-H(22A)	119.9	C(28)-C(29)-H(29A)	120.1
C(21)-C(22)-H(22A)	119.9	C(29)-C(30)-C(8)	121.16(19)
C(22)-C(23)-C(24)	120.1(2)	C(29)-C(30)-H(30A)	119.4
C(22)-C(23)-H(23A)	120.0	C(8)-C(30)-H(30A)	119.4
C(24)-C(23)-H(23A)	120.0		

Table S38. Anisotropic displacement parameters ($Å^2$ x 10^3) for (+/-)-**4**. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [h^2 a^{*2} U_{11} + ... + 2 h k a* b* U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	26(1)	30(1)	17(1)	2(1)	4(1)	1(1)
O2	21(1)	56(1)	30(1)	-2(1)	8(1)	-10(1)
O3	41(1)	26(1)	35(1)	-6(1)	1(1)	7(1)
O4	33(1)	30(1)	16(1)	-1(1)	4(1)	2(1)
N1	21(1)	31(1)	16(1)	1(1)	2(1)	1(1)
N2	23(1)	27(1)	18(1)	2(1)	4(1)	2(1)
C1	20(1)	25(1)	16(1)	1(1)	4(1)	0(1)
C2	20(1)	25(1)	15(1)	0(1)	3(1)	2(1)
C3	20(1)	21(1)	18(1)	-1(1)	5(1)	0(1)
C4	22(1)	22(1)	18(1)	-2(1)	4(1)	-1(1)
C5	18(1)	27(1)	21(1)	0(1)	6(1)	-4(1)
C6	17(1)	25(1)	20(1)	1(1)	6(1)	-1(1)
C7	29(1)	30(1)	12(1)	-1(1)	4(1)	-6(1)
C8	22(1)	26(1)	15(1)	3(1)	7(1)	-2(1)
C9	24(1)	21(1)	17(1)	-3(1)	3(1)	2(1)
C10	29(1)	24(1)	22(1)	-1(1)	10(1)	-4(1)
C11	21(1)	29(1)	28(1)	-6(1)	8(1)	-4(1)
C12	23(1)	26(1)	21(1)	-2(1)	2(1)	0(1)
C13	39(1)	32(1)	19(1)	4(1)	10(1)	1(1)
C14	22(1)	58(2)	36(1)	-8(1)	4(1)	-12(1)
C15	21(1)	26(1)	24(1)	4(1)	4(1)	0(1)
C16	24(1)	21(1)	34(1)	-3(1)	6(1)	-2(1)
C17	28(1)	28(1)	21(1)	-5(1)	3(1)	-3(1)
C18	22(1)	28(1)	20(1)	1(1)	5(1)	-2(1)
C19	52(2)	32(1)	49(2)	-7(1)	-6(1)	16(1)
C20	38(1)	33(1)	19(1)	-3(1)	4(1)	-1(1)
C21	42(1)	30(1)	21(1)	1(1)	9(1)	-1(1)
C22	67(2)	29(1)	26(1)	1(1)	14(1)	-10(1)
C23	63(2)	45(2)	21(1)	0(1)	10(1)	-29(1)
C24	35(1)	56(2)	30(1)	2(1)	9(1)	-19(1)
C25	29(1)	37(1)	28(1)	1(1)	6(1)	-6(1)
C26	22(1)	30(1)	26(1)	0(1)	4(1)	-2(1)
C27	26(1)	37(1)	29(1)	-4(1)	3(1)	-8(1)

C28	36(1)	31(1)	29(1)	-4(1)	7(1)	-8(1)
C29	31(1)	26(1)	24(1)	1(1)	8(1)	0(1)
C30	24(1)	30(1)	18(1)	0(1)	5(1)	-1(1)

Table S39. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for (+/-)-**4**.

	x	y	z	$U_{(eq)}$
H1	-225(19)	2477(18)	2914(11)	26(6)
H2	2720(20)	1043(19)	4855(12)	33(6)
H10A	-1257	1076	5527	29
H12A	-2265	2103	3502	28
H13A	1665	932	6614	44
H13B	519	240	6179	44
H13C	310	1501	6445	44
H14A	-4888	1186	4156	58
H14B	-3936	793	3662	58
H14C	-4101	2116	3818	58
H15A	3586	-679	4197	28
H17A	2211	-585	2104	31
H19A	4827	-2906	3394	69
H19B	3965	-2511	3937	69
H19C	5017	-1675	3748	69
H20A	502	1494	1075	45
H20B	838	233	1355	45
H20C	1930	1129	1316	45
H21A	881	4642	3154	37
H22A	2051	6098	2778	48
H23A	4054	5746	2579	51
H24A	4909	3938	2763	48
H25A	3757	2477	3153	37
H26A	4082	2897	5064	31
H27A	4783	4560	5665	37
H28A	3455	6117	5649	38
H29A	1400	5999	5046	32
H30A	665	4305	4508	28

Table S40. Torsion angles [°] for (+/-)-**4**.

C4-N1-C1-C6	81.54(19)	C2-C1-C6-C5	-13.71(19)
C4-N1-C1-C7	-149.01(16)	N1-C1-C6-C18	63.8(3)
C4-N1-C1-C2	-26.0(2)	C7-C1-C6-C18	-65.2(3)
C5-N2-C2-C3	84.11(19)	C2-C1-C6-C18	173.24(19)
C5-N2-C2-C8	-147.97(16)	N1-C1-C7-C25	-159.25(17)
C5-N2-C2-C1	-23.28(19)	C6-C1-C7-C25	-28.7(2)
N1-C1-C2-N2	140.09(15)	C2-C1-C7-C25	84.4(2)
C6-C1-C2-N2	21.69(17)	N1-C1-C7-C21	23.4(2)
C7-C1-C2-N2	-98.69(18)	C6-C1-C7-C21	153.97(18)
N1-C1-C2-C3	23.03(18)	C2-C1-C7-C21	-93.0(2)
C6-C1-C2-C3	-95.37(16)	N2-C2-C8-C26	7.8(2)
C7-C1-C2-C3	144.26(17)	C3-C2-C8-C26	136.18(18)
N1-C1-C2-C8	-97.40(18)	C1-C2-C8-C26	-110.4(2)
C6-C1-C2-C8	144.20(16)	N2-C2-C8-C30	-172.77(17)
C7-C1-C2-C8	23.8(2)	C3-C2-C8-C30	-44.3(2)
N2-C2-C3-C4	-123.37(18)	C1-C2-C8-C30	69.1(2)
C8-C2-C3-C4	108.95(19)	C13-O1-C9-C10	0.3(3)
C1-C2-C3-C4	-13.5(2)	C13-O1-C9-C3	-179.09(17)
N2-C2-C3-C9	57.1(3)	C4-C3-C9-O1	179.52(17)
C8-C2-C3-C9	-70.6(3)	C2-C3-C9-O1	-1.0(3)
C1-C2-C3-C9	166.9(2)	C4-C3-C9-C10	0.1(3)
C9-C3-C4-N1	177.58(18)	C2-C3-C9-C10	179.56(19)
C2-C3-C4-N1	-2.0(2)	O1-C9-C10-C11	-179.16(19)
C9-C3-C4-C12	-0.2(3)	C3-C9-C10-C11	0.2(3)
C2-C3-C4-C12	-179.75(18)	C14-O2-C11-C12	4.8(3)
C1-N1-C4-C3	18.7(2)	C14-O2-C11-C10	-175.20(19)
C1-N1-C4-C12	-163.7(2)	C9-C10-C11-O2	179.55(18)
C2-N2-C5-C6	15.6(2)	C9-C10-C11-C12	-0.5(3)
C2-N2-C5-C15	-170.55(18)	O2-C11-C12-C4	-179.66(19)
C15-C5-C6-C18	-0.2(3)	C10-C11-C12-C4	0.3(3)
N2-C5-C6-C18	173.83(17)	C3-C4-C12-C11	0.0(3)
C15-C5-C6-C1	-174.01(17)	N1-C4-C12-C11	-177.40(19)
N2-C5-C6-C1	0.0(2)	C6-C5-C15-C16	1.0(3)
N1-C1-C6-C5	-123.13(18)	N2-C5-C15-C16	-172.13(18)
C7-C1-C6-C5	107.89(18)	C19-O3-C16-C15	-9.8(3)

C19-O3-C16-C17	169.7(2)	C7-C21-C22-C23	0.9(3)
C5-C15-C16-O3	178.27(18)	C21-C22-C23-C24	-0.2(3)
C5-C15-C16-C17	-1.1(3)	C22-C23-C24-C25	-0.3(3)
O3-C16-C17-C18	-179.06(18)	C21-C7-C25-C24	0.8(3)
C15-C16-C17-C18	0.4(3)	C1-C7-C25-C24	-176.63(19)
C20-O4-C18-C17	-12.4(3)	C23-C24-C25-C7	0.0(3)
C20-O4-C18-C6	167.49(17)	C30-C8-C26-C27	0.8(3)
C16-C17-C18-O4	-179.54(18)	C2-C8-C26-C27	-179.75(18)
C16-C17-C18-C6	0.5(3)	C8-C26-C27-C28	-1.8(3)
C5-C6-C18-O4	179.43(17)	C26-C27-C28-C29	0.8(3)
C1-C6-C18-O4	-8.0(3)	C27-C28-C29-C30	1.4(3)
C5-C6-C18-C17	-0.6(3)	C28-C29-C30-C8	-2.5(3)
C1-C6-C18-C17	171.91(19)	C26-C8-C30-C29	1.4(3)
C25-C7-C21-C22	-1.2(3)	C2-C8-C30-C29	-178.10(18)
C1-C7-C21-C22	176.15(19)		

Table S41. Hydrogen bonds and close contacts for (+/-)-**4** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1...O4	0.91(2)	2.59(2)	3.158(2)	121.3(16)
N2-H2...O1	0.89(2)	2.48(2)	3.051(2)	122.3(18)
N2-H2...O2#1	0.89(2)	2.92(2)	3.635(2)	138.6(18)

Symmetry transformations used to generate equivalent atoms:

#1 -x, -y, -z-1

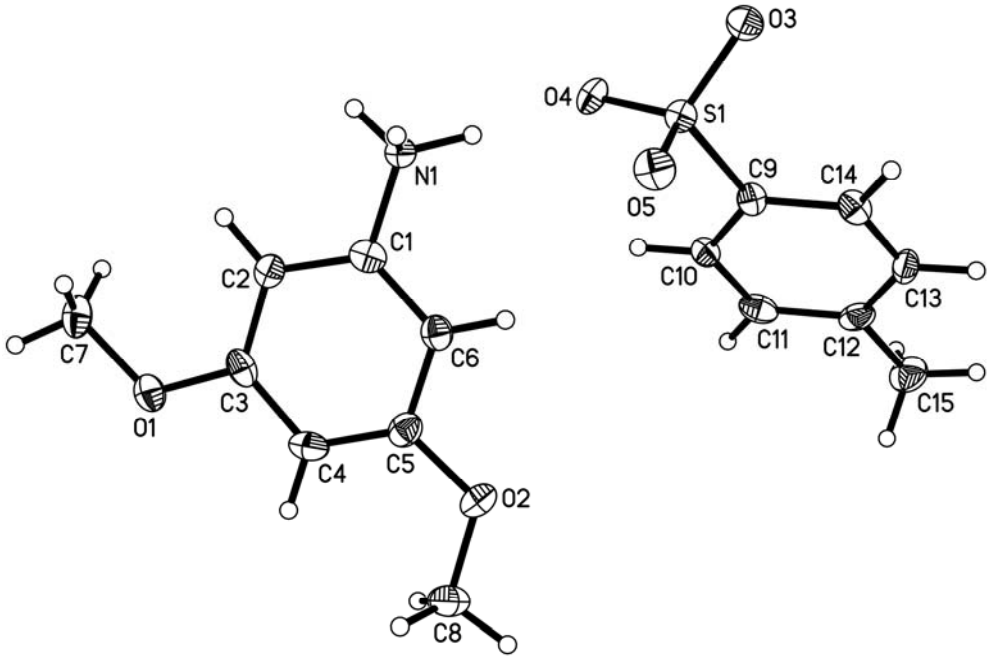


Figure S60. ORTEP diagram of **5** with thermal ellipsoids drawn at 50% probability level.

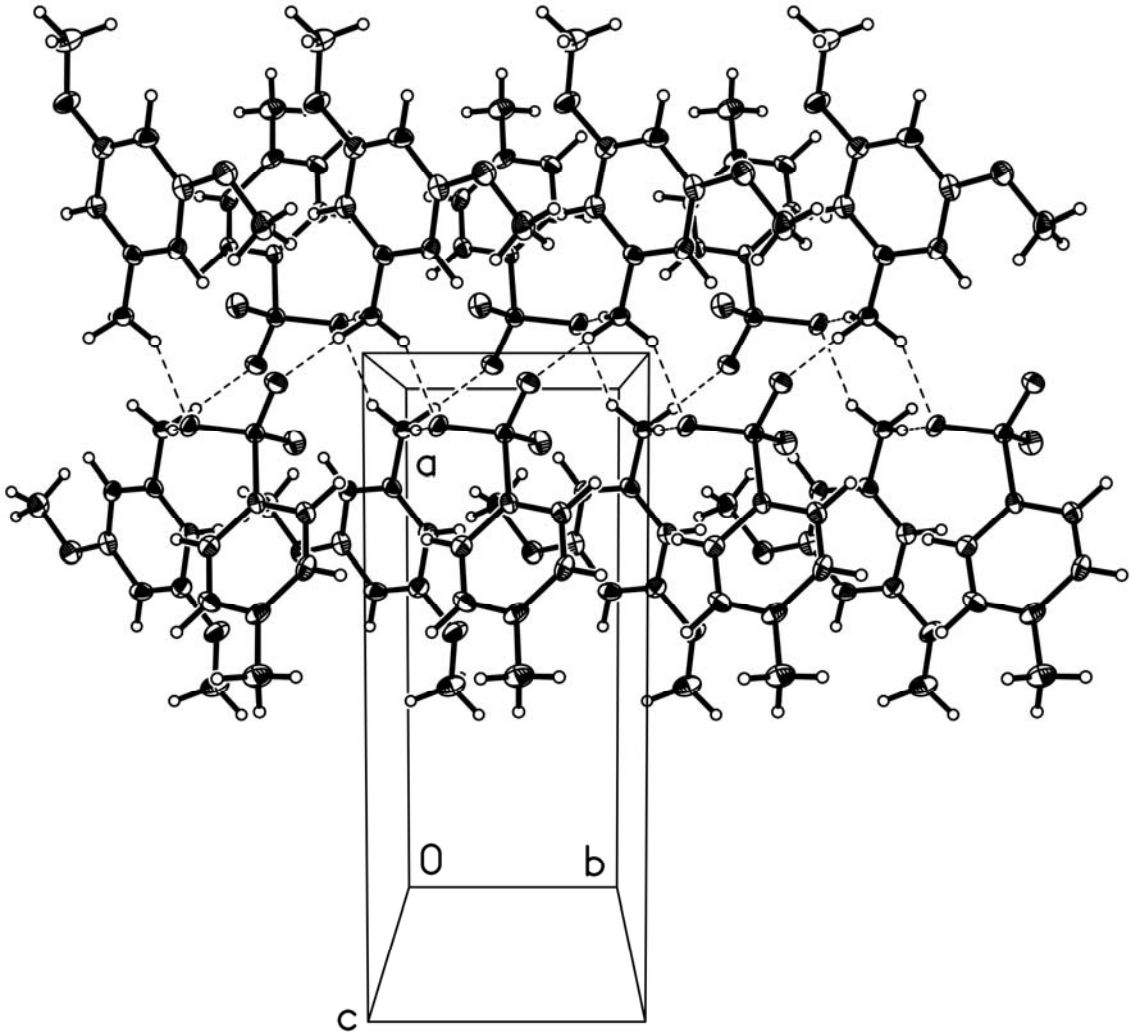


Figure S61. ORTEP diagram of **5** with thermal ellipsoids drawn at 50% probability level showing intermolecular hydrogen bonding.

Experimental Details for the Structure Determination of **5**.

Data collection

A crystal (0.38 x 0.15 x 0.06 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 120 seconds and a detector distance of 4.01 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 2539 strong reflections from the actual data collection after integration.[3] See Table S42 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group $P2_1/n$ was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. Ammonium hydrogen atoms were found from the difference Fourier map, and their positional and isotropic displacement parameters were refined independently from those of the nitrogen atom. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters.

The refinement stalled at $R1 = 0.139$, at which point twin modeling was required. After the non-merohedral twin law, [0.571 0 -0.429 / 0 -1 0 / -1.571 0 -0.571], a 180º rotation about reciprocal lattice [-1 0 1], was determined,[6] the data were re-integrated,[3] and a new absorption correction was applied.[7] There were 2074 unique reflections solely in the first component, 1960 unique reflections solely in the second component, and 1390 unique overlapping reflections. The mass ratio of the two components refined to 60:40. The final full matrix least squares refinement converged to $R1 = 0.0528$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1117$ (F^2, all data).

Structure description

The structure is the one suggested, with all atoms in general positions. Moderate intermolecular hydrogen bonding links the molecules one-dimensionally in the direction parallel to the *b*-axis (see Figure S61 and Table S48).

References

[1] *APEX2*, version 2011.4-1; Bruker AXS: Madison, WI, 2011.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.68A; Bruker AXS: Madison, WI, 2009.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008,** *A64*, 112-122.

[6] a) Parsons, S.; Gould, B.; Cooper, R.; Farrugia, L. *ROTAX*; University of Edinburgh: Edinburgh, Scotland, 2003; b) Sheldrick, G. M. *CELL_NOW: A program that analyzes a list of reflections to find a cell and orientation matrix despite the presence of several twin domains or other junk*, version 2008/2; University of Göttingen: Göttingen, Germany, 2008.

[7] Sheldrick, G. M. *TWINABS*, version 2008/4; University of Göttingen: Göttingen, Germany, 2008.

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma|F_o^2|$$

$$R1 = \Sigma||F_o| - |F_c|| \,/\, \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max(0, F_o^2) + 2/3\, F_c^2$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

Table S42. Crystal data and structure refinement for **5**.

Identification code	**5**, JONJK70	
Empirical formula	C15 H19 N O5 S	
Formula weight	325.37	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 13.031(4) Å	α = 90°
	b = 5.3646(17) Å	β = 99.179(6)°
	c = 22.323(7) Å	γ = 90°
Volume	1540.6(8) Å^3	
Z	4	
Density (calculated)	1.403 Mg/m^3	
Absorption coefficient	0.233 mm^{-1}	
F(000)	688	
Crystal color, morphology	colorless, needle	
Crystal size	0.38 x 0.15 x 0.06 mm^3	
Theta range for data collection	1.85 to 25.03°	
Index ranges	$-15 \le h \le 15$, $0 \le k \le 6$, $0 \le l \le 26$	
Reflections collected	24078	
Independent reflections	2727 [R(int) = 0.1286]	
Observed reflections	1902	
Completeness to theta = 25.03°	99.8%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9861 and 0.9166	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2727 / 0 / 215	
Goodness-of-fit on F^2	1.041	
Final R indices [I>2sigma(I)]	R1 = 0.0528, wR2 = 0.0970	
R indices (all data)	R1 = 0.0945, wR2 = 0.1117	
Largest diff. peak and hole	0.312 and -0.393 e.Å^{-3}	

Table S43. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	6877(2)	14063(5)	6667(1)	26(1)
O2	5510(2)	7315(5)	5408(1)	28(1)
N1	9056(2)	9453(7)	5471(2)	19(1)
C1	8057(3)	9968(7)	5664(2)	19(1)
C2	7996(3)	11911(7)	6066(2)	18(1)
C3	7048(3)	12257(7)	6262(2)	21(1)
C4	6199(3)	10758(7)	6052(2)	23(1)
C5	6291(3)	8867(7)	5652(2)	21(1)
C6	7238(3)	8428(7)	5456(2)	19(1)
C7	7739(3)	15587(8)	6918(2)	27(1)
C8	4544(3)	7518(9)	5634(2)	31(1)
S1	8954(1)	5190(2)	4166(1)	18(1)
O3	9783(2)	4230(5)	3860(1)	24(1)
O4	9086(2)	7854(4)	4293(1)	22(1)
O5	8806(2)	3768(5)	4697(1)	24(1)
C9	7802(3)	4991(7)	3633(2)	17(1)
C10	7067(3)	6879(7)	3593(2)	20(1)
C11	6170(3)	6727(8)	3165(2)	22(1)
C12	5996(3)	4698(8)	2770(2)	22(1)
C13	6745(3)	2833(7)	2823(2)	21(1)
C14	7634(3)	2948(7)	3254(2)	20(1)
C15	5037(3)	4588(9)	2302(2)	30(1)

Table S44. Bond lengths [Å] and angles [°] for **5**.

O(1)-C(3)	1.367(4)	C(13)-C(14)	1.386(5)
O(1)-C(7)	1.430(4)	C(13)-H(13A)	0.9500
O(2)-C(5)	1.359(4)	C(14)-H(14A)	0.9500
O(2)-C(8)	1.433(4)	C(15)-H(15A)	0.9800
N(1)-C(1)	1.462(5)	C(15)-H(15B)	0.9800
N(1)-H(1N)	1.00(4)	C(15)-H(15C)	0.9800
N(1)-H(2N)	0.96(5)	C(3)-O(1)-C(7)	117.7(3)
N(1)-H(3N)	0.90(4)	C(5)-O(2)-C(8)	117.2(3)
C(1)-C(6)	1.371(5)	C(1)-N(1)-H(1N)	116(2)
C(1)-C(2)	1.385(5)	C(1)-N(1)-H(2N)	112(3)
C(2)-C(3)	1.387(5)	H(1N)-N(1)-H(2N)	104(3)
C(2)-H(2A)	0.9500	C(1)-N(1)-H(3N)	110(3)
C(3)-C(4)	1.387(5)	H(1N)-N(1)-H(3N)	114(3)
C(4)-C(5)	1.369(6)	H(2N)-N(1)-H(3N)	99(3)
C(4)-H(4A)	0.9500	C(6)-C(1)-C(2)	123.4(3)
C(5)-C(6)	1.393(5)	C(6)-C(1)-N(1)	117.9(3)
C(6)-H(6A)	0.9500	C(2)-C(1)-N(1)	118.7(3)
C(7)-H(7A)	0.9800	C(1)-C(2)-C(3)	116.8(3)
C(7)-H(7B)	0.9800	C(1)-C(2)-H(2A)	121.6
C(7)-H(7C)	0.9800	C(3)-C(2)-H(2A)	121.6
C(8)-H(8A)	0.9800	O(1)-C(3)-C(4)	115.4(3)
C(8)-H(8B)	0.9800	O(1)-C(3)-C(2)	123.4(4)
C(8)-H(8C)	0.9800	C(4)-C(3)-C(2)	121.2(4)
S(1)-O(5)	1.448(3)	C(5)-C(4)-C(3)	120.1(4)
S(1)-O(3)	1.461(3)	C(5)-C(4)-H(4A)	119.9
S(1)-O(4)	1.462(3)	C(3)-C(4)-H(4A)	119.9
S(1)-C(9)	1.764(4)	O(2)-C(5)-C(4)	125.2(3)
C(9)-C(14)	1.380(5)	O(2)-C(5)-C(6)	114.5(3)
C(9)-C(10)	1.387(5)	C(4)-C(5)-C(6)	120.3(4)
C(10)-C(11)	1.389(5)	C(1)-C(6)-C(5)	118.2(4)
C(10)-H(10A)	0.9500	C(1)-C(6)-H(6A)	120.9
C(11)-C(12)	1.397(5)	C(5)-C(6)-H(6A)	120.9
C(11)-H(11A)	0.9500	O(1)-C(7)-H(7A)	109.5
C(12)-C(13)	1.389(5)	O(1)-C(7)-H(7B)	109.5
C(12)-C(15)	1.497(5)	H(7A)-C(7)-H(7B)	109.5

O(1)-C(7)-H(7C)	109.5	C(11)-C(10)-H(10A)	120.1
H(7A)-C(7)-H(7C)	109.5	C(10)-C(11)-C(12)	120.9(4)
H(7B)-C(7)-H(7C)	109.5	C(10)-C(11)-H(11A)	119.5
O(2)-C(8)-H(8A)	109.5	C(12)-C(11)-H(11A)	119.5
O(2)-C(8)-H(8B)	109.5	C(13)-C(12)-C(11)	117.9(3)
H(8A)-C(8)-H(8B)	109.5	C(13)-C(12)-C(15)	121.8(4)
O(2)-C(8)-H(8C)	109.5	C(11)-C(12)-C(15)	120.3(4)
H(8A)-C(8)-H(8C)	109.5	C(14)-C(13)-C(12)	121.7(4)
H(8B)-C(8)-H(8C)	109.5	C(14)-C(13)-H(13A)	119.2
O(5)-S(1)-O(3)	113.71(16)	C(12)-C(13)-H(13A)	119.2
O(5)-S(1)-O(4)	112.49(16)	C(9)-C(14)-C(13)	119.6(4)
O(3)-S(1)-O(4)	111.31(16)	C(9)-C(14)-H(14A)	120.2
O(5)-S(1)-C(9)	108.06(16)	C(13)-C(14)-H(14A)	120.2
O(3)-S(1)-C(9)	106.02(16)	C(12)-C(15)-H(15A)	109.5
O(4)-S(1)-C(9)	104.56(16)	C(12)-C(15)-H(15B)	109.5
C(14)-C(9)-C(10)	120.0(3)	H(15A)-C(15)-H(15B)	109.5
C(14)-C(9)-S(1)	119.7(3)	C(12)-C(15)-H(15C)	109.5
C(10)-C(9)-S(1)	120.2(3)	H(15A)-C(15)-H(15C)	109.5
C(9)-C(10)-C(11)	119.9(4)	H(15B)-C(15)-H(15C)	109.5
C(9)-C(10)-H(10A)	120.1		

Table S45. Anisotropic displacement parameters ($Å^2$ x 10^3) for **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [h^2 a^{*2} U_{11} + ... + 2 h k a* b* U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	28(2)	24(2)	26(2)	-8(1)	8(1)	1(1)
O2	21(2)	31(2)	32(2)	-7(1)	3(1)	-9(1)
N1	16(2)	20(2)	20(2)	-2(2)	4(1)	-1(2)
C1	20(2)	19(2)	20(2)	6(2)	4(2)	4(2)
C2	22(2)	16(2)	15(2)	3(2)	1(2)	-2(2)
C3	30(2)	19(2)	15(2)	1(2)	7(2)	4(2)
C4	17(2)	30(2)	24(2)	5(2)	7(2)	1(2)
C5	21(2)	22(2)	19(2)	2(2)	0(2)	-2(2)
C6	23(2)	19(2)	16(2)	1(2)	3(2)	1(2)
C7	33(2)	22(2)	24(2)	-7(2)	1(2)	0(2)
C8	23(2)	42(3)	31(3)	-1(2)	9(2)	-7(2)
S1	19(1)	18(1)	18(1)	0(1)	2(1)	0(1)
O3	19(1)	24(2)	28(2)	-2(1)	3(1)	3(1)
O4	23(1)	17(1)	23(2)	-2(1)	-1(1)	-5(1)
O5	29(2)	25(2)	18(2)	8(1)	3(1)	1(1)
C9	20(2)	14(2)	16(2)	2(2)	3(2)	2(2)
C10	21(2)	21(2)	18(2)	-2(2)	7(2)	-2(2)
C11	19(2)	24(2)	25(2)	8(2)	8(2)	6(2)
C12	20(2)	27(2)	20(2)	4(2)	5(2)	-8(2)
C13	28(2)	17(2)	18(2)	-4(2)	6(2)	-6(2)
C14	23(2)	18(2)	20(2)	2(2)	6(2)	3(2)
C15	23(2)	38(3)	26(2)	-1(2)	-3(2)	-4(2)

Table S46. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **5**.

	x	y	z	$U_{(eq)}$
H1N	9030(30)	8860(70)	5040(20)	28(11)
H2N	9440(40)	8170(90)	5720(20)	53(15)
H3N	9490(30)	10740(80)	5569(18)	26(12)
H2A	8575	12956	6201	22
H4A	5553	11044	6186	28
H6A	7314	7099	5185	23
H7A	7521	16746	7213	40
H7B	7984	16532	6592	40
H7C	8302	14528	7120	40
H8A	4043	6330	5420	47
H8B	4272	9216	5566	47
H8C	4656	7148	6069	47
H10A	7178	8274	3858	23
H11A	5667	8020	3141	27
H13A	6644	1444	2556	25
H14A	8126	1627	3289	24
H15A	5018	6035	2033	45
H15B	4422	4601	2503	45
H15C	5044	3055	2064	45

Table S47. Torsion angles [°] for **5**.

C6-C1-C2-C3	0.5(6)	O5-S1-C9-C14	-83.0(3)
N1-C1-C2-C3	-176.8(3)	O3-S1-C9-C14	39.2(3)
C7-O1-C3-C4	177.3(3)	O4-S1-C9-C14	156.9(3)
C7-O1-C3-C2	-2.9(5)	O5-S1-C9-C10	97.0(3)
C1-C2-C3-O1	178.7(3)	O3-S1-C9-C10	-140.7(3)
C1-C2-C3-C4	-1.5(6)	O4-S1-C9-C10	-23.0(3)
O1-C3-C4-C5	-179.0(3)	C14-C9-C10-C11	-1.0(5)
C2-C3-C4-C5	1.2(6)	S1-C9-C10-C11	179.0(3)
C8-O2-C5-C4	-6.5(6)	C9-C10-C11-C12	-0.4(5)
C8-O2-C5-C6	174.4(3)	C10-C11-C12-C13	0.6(5)
C3-C4-C5-O2	-178.8(4)	C10-C11-C12-C15	-178.6(4)
C3-C4-C5-C6	0.3(6)	C11-C12-C13-C14	0.4(5)
C2-C1-C6-C5	1.0(6)	C15-C12-C13-C14	179.6(4)
N1-C1-C6-C5	178.2(3)	C10-C9-C14-C13	2.0(5)
O2-C5-C6-C1	177.9(3)	S1-C9-C14-C13	-177.9(3)
C4-C5-C6-C1	-1.4(6)	C12-C13-C14-C9	-1.8(6)

Table S48. Hydrogen bonds and close contacts for **5** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1N...O4	1.00(4)	1.78(4)	2.772(4)	176(3)
N1-H2N...O3#1	0.96(5)	1.81(5)	2.775(4)	176(4)
N1-H3N...O4#2	0.90(4)	1.98(4)	2.796(4)	150(4)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y+1,-z+1 #2 -x+2,-y+2,-z+1

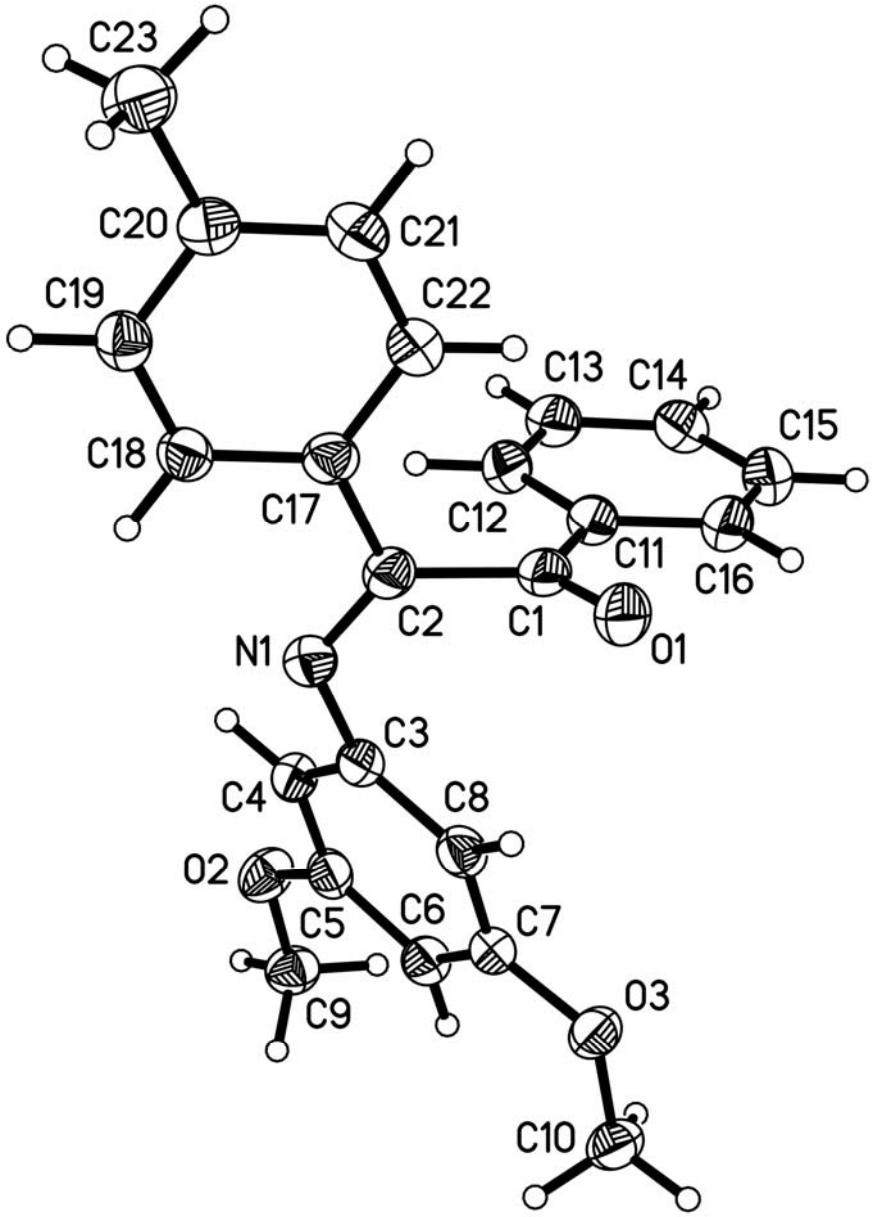


Figure S62. ORTEP diagram of **6b** with thermal ellipsoids drawn at 50% probability level.

Experimental Details for the Structure Determination of **6b**.

Data collection

A crystal (0.32 x 0.28 x 0.08 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 3.98 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 3346 strong reflections from the actual data collection after integration.[3] See Table S49 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group $P2_1/c$ was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1$ = 0.0619 (F^2, $I > 2\sigma(I)$) and $wR2$ = 0.1470 (F^2, all data).

Structure description

The structure is the one suggested with all atoms in general positions.

References

[1] *APEX2*, version 2011.4-1; Bruker AXS: Madison, WI, 2011.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.68A; Bruker AXS: Madison, WI, 2009.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma|F_o^2|$$

$$R1 = \Sigma||F_o| - |F_c|| \,/\, \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max(0, F_o^2) + 2/3\, F_c^2$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] / (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

Table S49. Crystal data and structure refinement for **6b**.

Identification code	**6b**, JONJK62	
Empirical formula	C23 H21 N O3	
Formula weight	359.41	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 9.0505(18) Å	α = 90°
	b = 23.669(5) Å	β = 97.838(4)°
	c = 8.6237(18) Å	γ = 90°
Volume	1830.0(6) Å^3	
Z	4	
Density (calculated)	1.304 Mg/m^3	
Absorption coefficient	0.086 mm^{-1}	
F(000)	760	
Crystal color, morphology	pale yellow, plate	
Crystal size	0.32 x 0.28 x 0.08 mm^3	
Theta range for data collection	1.72 to 27.48°	
Index ranges	$-11 \le h \le 11$, $-30 \le k \le 30$, $-11 \le l \le 11$	
Reflections collected	21409	
Independent reflections	4191 [R(int) = 0.0942]	
Observed reflections	2558	
Completeness to theta = 27.48°	99.9%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9931 and 0.9729	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4191 / 0 / 247	
Goodness-of-fit on F^2	1.029	
Final R indices [I>2sigma(I)]	R1 = 0.0619, wR2 = 0.1275	
R indices (all data)	R1 = 0.1121, wR2 = 0.1470	
Largest diff. peak and hole	0.480 and -0.307 e.Å^{-3}	

Table S50. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **6b**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	3386(2)	1548(1)	9184(2)	37(1)
O2	2153(2)	-864(1)	4586(2)	35(1)
O3	6260(2)	-15(1)	8013(2)	32(1)
N1	1171(2)	459(1)	8394(2)	29(1)
C1	2414(3)	1381(1)	8164(3)	31(1)
C2	1201(3)	992(1)	8626(3)	28(1)
C3	2279(3)	180(1)	7658(3)	26(1)
C4	1757(3)	-198(1)	6468(3)	28(1)
C5	2768(3)	-515(1)	5771(3)	28(1)
C6	4295(3)	-476(1)	6255(3)	29(1)
C7	4789(3)	-99(1)	7459(3)	28(1)
C8	3796(3)	228(1)	8156(3)	28(1)
C9	3131(3)	-1227(1)	3881(3)	33(1)
C10	7316(3)	-402(1)	7527(3)	34(1)
C11	2334(3)	1549(1)	6506(3)	31(1)
C12	1137(3)	1391(1)	5406(3)	33(1)
C13	1070(3)	1565(1)	3865(3)	36(1)
C14	2206(3)	1893(1)	3400(4)	38(1)
C15	3396(3)	2054(1)	4490(4)	39(1)
C16	3462(3)	1885(1)	6037(4)	37(1)
C17	2(3)	1268(1)	9376(3)	28(1)
C18	-1108(3)	941(1)	9915(3)	29(1)
C19	-2242(3)	1193(1)	10572(3)	30(1)
C20	-2303(3)	1778(1)	10738(3)	30(1)
C21	-1212(3)	2102(1)	10188(3)	35(1)
C22	-74(3)	1852(1)	9517(3)	32(1)
C23	-3486(3)	2042(1)	11557(4)	44(1)

Table S51. Bond lengths [Å] and angles [°] for **6b**.

O(1)-C(1)	1.223(3)	C(16)-H(16A)	0.9500
O(2)-C(5)	1.372(3)	C(17)-C(22)	1.390(3)
O(2)-C(9)	1.427(3)	C(17)-C(18)	1.396(3)
O(3)-C(7)	1.366(3)	C(18)-C(19)	1.375(3)
O(3)-C(10)	1.427(3)	C(18)-H(18A)	0.9500
N(1)-C(2)	1.276(3)	C(19)-C(20)	1.393(3)
N(1)-C(3)	1.420(3)	C(19)-H(19A)	0.9500
C(1)-C(11)	1.477(4)	C(20)-C(21)	1.384(4)
C(1)-C(2)	1.526(3)	C(20)-C(23)	1.498(4)
C(2)-C(17)	1.488(3)	C(21)-C(22)	1.383(4)
C(3)-C(8)	1.386(3)	C(21)-H(21A)	0.9500
C(3)-C(4)	1.395(3)	C(22)-H(22A)	0.9500
C(4)-C(5)	1.382(3)	C(23)-H(23A)	0.9800
C(4)-H(4A)	0.9500	C(23)-H(23B)	0.9800
C(5)-C(6)	1.392(3)	C(23)-H(23C)	0.9800
C(6)-C(7)	1.396(3)	C(5)-O(2)-C(9)	117.89(19)
C(6)-H(6A)	0.9500	C(7)-O(3)-C(10)	117.70(19)
C(7)-C(8)	1.384(3)	C(2)-N(1)-C(3)	121.9(2)
C(8)-H(8A)	0.9500	O(1)-C(1)-C(11)	123.0(2)
C(9)-H(9A)	0.9800	O(1)-C(1)-C(2)	118.7(2)
C(9)-H(9B)	0.9800	C(11)-C(1)-C(2)	118.3(2)
C(9)-H(9C)	0.9800	N(1)-C(2)-C(17)	120.0(2)
C(10)-H(10A)	0.9800	N(1)-C(2)-C(1)	123.7(2)
C(10)-H(10B)	0.9800	C(17)-C(2)-C(1)	116.3(2)
C(10)-H(10C)	0.9800	C(8)-C(3)-C(4)	120.1(2)
C(11)-C(12)	1.390(4)	C(8)-C(3)-N(1)	123.6(2)
C(11)-C(16)	1.397(4)	C(4)-C(3)-N(1)	116.0(2)
C(12)-C(13)	1.385(4)	C(5)-C(4)-C(3)	119.4(2)
C(12)-H(12A)	0.9500	C(5)-C(4)-H(4A)	120.3
C(13)-C(14)	1.390(4)	C(3)-C(4)-H(4A)	120.3
C(13)-H(13A)	0.9500	O(2)-C(5)-C(4)	115.2(2)
C(14)-C(15)	1.383(4)	O(2)-C(5)-C(6)	123.3(2)
C(14)-H(14A)	0.9500	C(4)-C(5)-C(6)	121.5(2)
C(15)-C(16)	1.385(4)	C(5)-C(6)-C(7)	118.0(2)
C(15)-H(15A)	0.9500	C(5)-C(6)-H(6A)	121.0

C(7)-C(6)-H(6A)	121.0	C(14)-C(15)-C(16)	120.2(3)
O(3)-C(7)-C(8)	115.3(2)	C(14)-C(15)-H(15A)	119.9
O(3)-C(7)-C(6)	123.4(2)	C(16)-C(15)-H(15A)	119.9
C(8)-C(7)-C(6)	121.3(2)	C(15)-C(16)-C(11)	120.3(3)
C(7)-C(8)-C(3)	119.6(2)	C(15)-C(16)-H(16A)	119.8
C(7)-C(8)-H(8A)	120.2	C(11)-C(16)-H(16A)	119.8
C(3)-C(8)-H(8A)	120.2	C(22)-C(17)-C(18)	118.4(2)
O(2)-C(9)-H(9A)	109.5	C(22)-C(17)-C(2)	121.4(2)
O(2)-C(9)-H(9B)	109.5	C(18)-C(17)-C(2)	120.1(2)
H(9A)-C(9)-H(9B)	109.5	C(19)-C(18)-C(17)	120.5(2)
O(2)-C(9)-H(9C)	109.5	C(19)-C(18)-H(18A)	119.8
H(9A)-C(9)-H(9C)	109.5	C(17)-C(18)-H(18A)	119.8
H(9B)-C(9)-H(9C)	109.5	C(18)-C(19)-C(20)	121.1(2)
O(3)-C(10)-H(10A)	109.5	C(18)-C(19)-H(19A)	119.4
O(3)-C(10)-H(10B)	109.5	C(20)-C(19)-H(19A)	119.4
H(10A)-C(10)-H(10B)	109.5	C(21)-C(20)-C(19)	118.4(2)
O(3)-C(10)-H(10C)	109.5	C(21)-C(20)-C(23)	121.3(2)
H(10A)-C(10)-H(10C)	109.5	C(19)-C(20)-C(23)	120.3(2)
H(10B)-C(10)-H(10C)	109.5	C(22)-C(21)-C(20)	120.8(2)
C(12)-C(11)-C(16)	119.2(3)	C(22)-C(21)-H(21A)	119.6
C(12)-C(11)-C(1)	121.3(2)	C(20)-C(21)-H(21A)	119.6
C(16)-C(11)-C(1)	119.5(2)	C(21)-C(22)-C(17)	120.7(2)
C(13)-C(12)-C(11)	120.2(3)	C(21)-C(22)-H(22A)	119.6
C(13)-C(12)-H(12A)	119.9	C(17)-C(22)-H(22A)	119.6
C(11)-C(12)-H(12A)	119.9	C(20)-C(23)-H(23A)	109.5
C(12)-C(13)-C(14)	120.4(3)	C(20)-C(23)-H(23B)	109.5
C(12)-C(13)-H(13A)	119.8	H(23A)-C(23)-H(23B)	109.5
C(14)-C(13)-H(13A)	119.8	C(20)-C(23)-H(23C)	109.5
C(15)-C(14)-C(13)	119.7(3)	H(23A)-C(23)-H(23C)	109.5
C(15)-C(14)-H(14A)	120.2	H(23B)-C(23)-H(23C)	109.5
C(13)-C(14)-H(14A)	120.2		

Table S52. Anisotropic displacement parameters ($Å^2$ x 10^3) for **6b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [h^2 a^{*2} U_{11} + ... + 2 h k a* b* U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	32(1)	33(1)	45(1)	-5(1)	-1(1)	-5(1)
O2	28(1)	39(1)	36(1)	-9(1)	0(1)	-1(1)
O3	24(1)	30(1)	42(1)	-6(1)	2(1)	0(1)
N1	28(1)	28(1)	31(1)	1(1)	2(1)	1(1)
C1	25(1)	24(1)	44(2)	-4(1)	3(1)	3(1)
C2	24(1)	28(1)	29(1)	-1(1)	0(1)	0(1)
C3	25(1)	23(1)	32(1)	3(1)	8(1)	2(1)
C4	23(1)	28(1)	32(1)	4(1)	2(1)	-2(1)
C5	30(1)	25(1)	28(1)	3(1)	3(1)	-4(1)
C6	28(1)	27(1)	31(1)	2(1)	6(1)	1(1)
C7	25(1)	24(1)	35(2)	5(1)	2(1)	-1(1)
C8	30(1)	24(1)	30(1)	1(1)	0(1)	-3(1)
C9	32(2)	32(1)	34(2)	-4(1)	2(1)	3(1)
C10	26(1)	35(1)	40(2)	-2(1)	2(1)	4(1)
C11	27(1)	25(1)	40(2)	-2(1)	6(1)	0(1)
C12	29(1)	26(1)	44(2)	-3(1)	5(1)	-2(1)
C13	34(2)	32(1)	43(2)	-2(1)	3(1)	2(1)
C14	39(2)	29(1)	47(2)	2(1)	8(1)	2(1)
C15	35(2)	33(1)	52(2)	4(1)	11(1)	-4(1)
C16	28(1)	33(1)	49(2)	-3(1)	3(1)	-3(1)
C17	26(1)	28(1)	29(1)	1(1)	-2(1)	2(1)
C18	29(1)	26(1)	31(1)	1(1)	0(1)	0(1)
C19	30(1)	31(1)	27(1)	3(1)	2(1)	-2(1)
C20	30(1)	32(1)	27(1)	0(1)	2(1)	2(1)
C21	39(2)	24(1)	42(2)	0(1)	5(1)	1(1)
C22	33(1)	29(1)	36(2)	3(1)	6(1)	0(1)
C23	39(2)	43(2)	50(2)	-1(1)	5(1)	4(1)

Table S53. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **6b**.

	x	y	z	$U_{(eq)}$
H4A	717	-238	6141	33
H6A	4981	-699	5780	34
H8A	4152	484	8972	34
H9A	2548	-1461	3084	50
H9B	3675	-1470	4684	50
H9C	3842	-998	3391	50
H10A	8314	-311	8060	51
H10B	7309	-373	6393	51
H10C	7051	-788	7795	51
H12A	363	1164	5713	40
H13A	243	1460	3121	44
H14A	2166	2006	2338	46
H15A	4171	2280	4178	47
H16A	4279	1999	6783	44
H18A	-1079	541	9828	34
H19A	-2997	965	10918	36
H21A	-1247	2502	10274	42
H22A	665	2081	9147	39
H23A	-4469	1914	11067	66
H23B	-3426	2454	11478	66
H23C	-3335	1931	12662	66

Table S54. Torsion angles [°] for **6b**.

C3-N1-C2-C17	178.9(2)	C2-C1-C11-C12	4.3(3)
C3-N1-C2-C1	-0.6(4)	O1-C1-C11-C16	4.3(4)
O1-C1-C2-N1	-103.0(3)	C2-C1-C11-C16	-177.4(2)
C11-C1-C2-N1	78.5(3)	C16-C11-C12-C13	0.2(4)
O1-C1-C2-C17	77.4(3)	C1-C11-C12-C13	178.5(2)
C11-C1-C2-C17	-101.0(3)	C11-C12-C13-C14	0.7(4)
C2-N1-C3-C8	53.5(3)	C12-C13-C14-C15	-1.1(4)
C2-N1-C3-C4	-132.6(2)	C13-C14-C15-C16	0.5(4)
C8-C3-C4-C5	-1.4(4)	C14-C15-C16-C11	0.5(4)
N1-C3-C4-C5	-175.5(2)	C12-C11-C16-C15	-0.8(4)
C9-O2-C5-C4	-176.4(2)	C1-C11-C16-C15	-179.1(2)
C9-O2-C5-C6	3.6(3)	N1-C2-C17-C22	-174.0(2)
C3-C4-C5-O2	-178.3(2)	C1-C2-C17-C22	5.5(4)
C3-C4-C5-C6	1.7(4)	N1-C2-C17-C18	4.0(4)
O2-C5-C6-C7	179.0(2)	C1-C2-C17-C18	-176.5(2)
C4-C5-C6-C7	-0.9(4)	C22-C17-C18-C19	-0.2(4)
C10-O3-C7-C8	170.0(2)	C2-C17-C18-C19	-178.3(2)
C10-O3-C7-C6	-11.3(3)	C17-C18-C19-C20	-1.0(4)
C5-C6-C7-O3	-178.7(2)	C18-C19-C20-C21	1.6(4)
C5-C6-C7-C8	0.0(4)	C18-C19-C20-C23	-175.7(2)
O3-C7-C8-C3	179.0(2)	C19-C20-C21-C22	-1.2(4)
C6-C7-C8-C3	0.2(4)	C23-C20-C21-C22	176.1(3)
C4-C3-C8-C7	0.5(4)	C20-C21-C22-C17	0.0(4)
N1-C3-C8-C7	174.1(2)	C18-C17-C22-C21	0.6(4)
O1-C1-C11-C12	-174.0(2)	C2-C17-C22-C21	178.7(2)

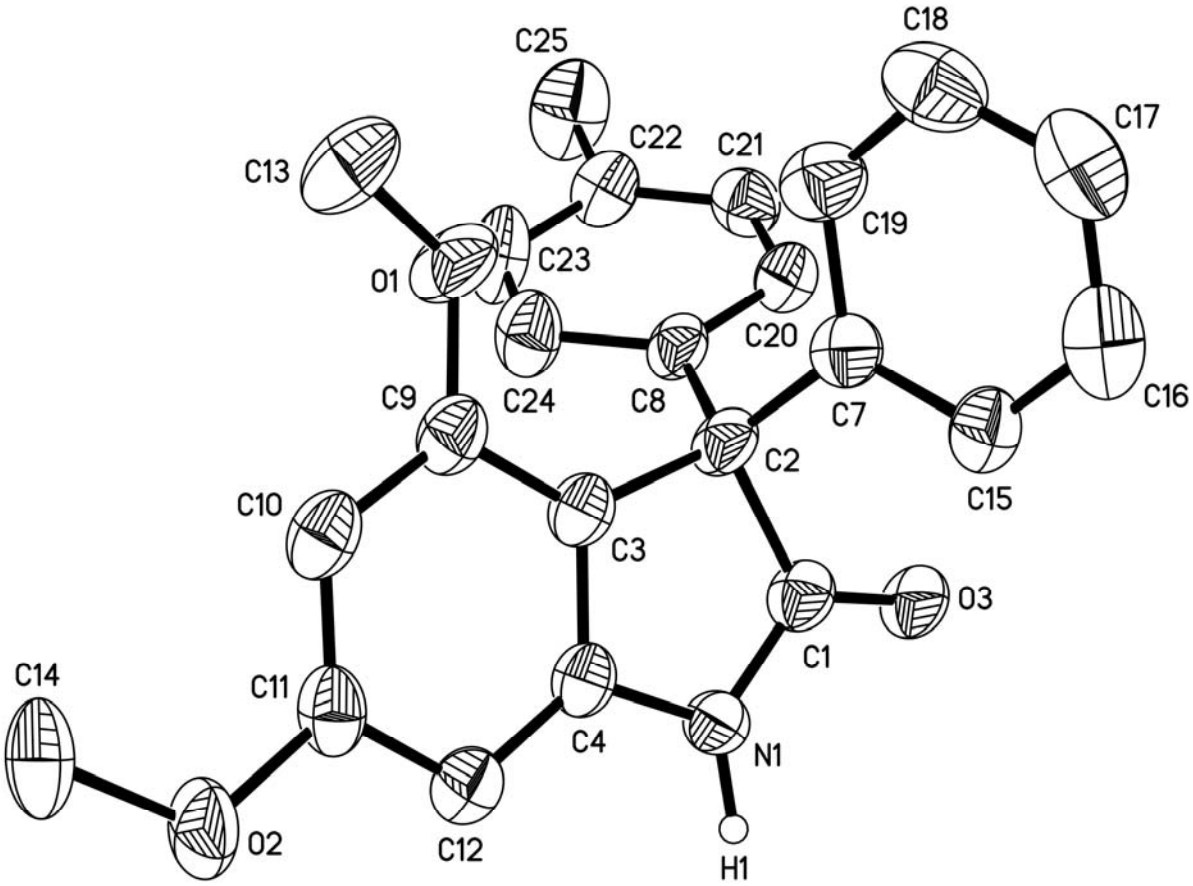


Figure S63. ORTEP diagram of **7** with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1, to emphasize valency of the nitrogen atom.

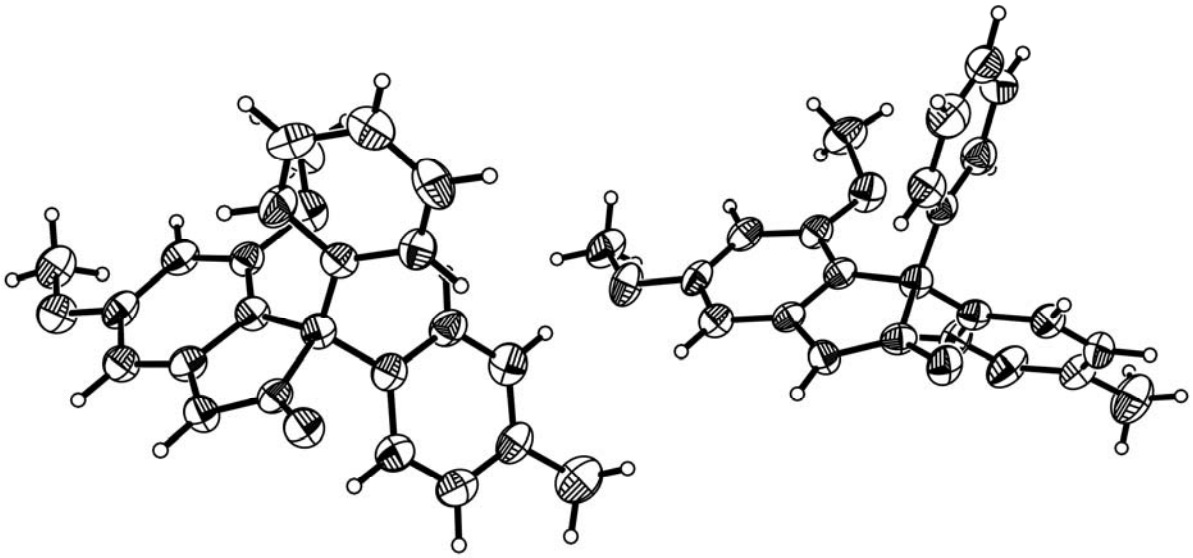


Figure S64. ORTEP diagram of asymmetric unit of **7** with thermal ellipsoids drawn at 50% probability level.

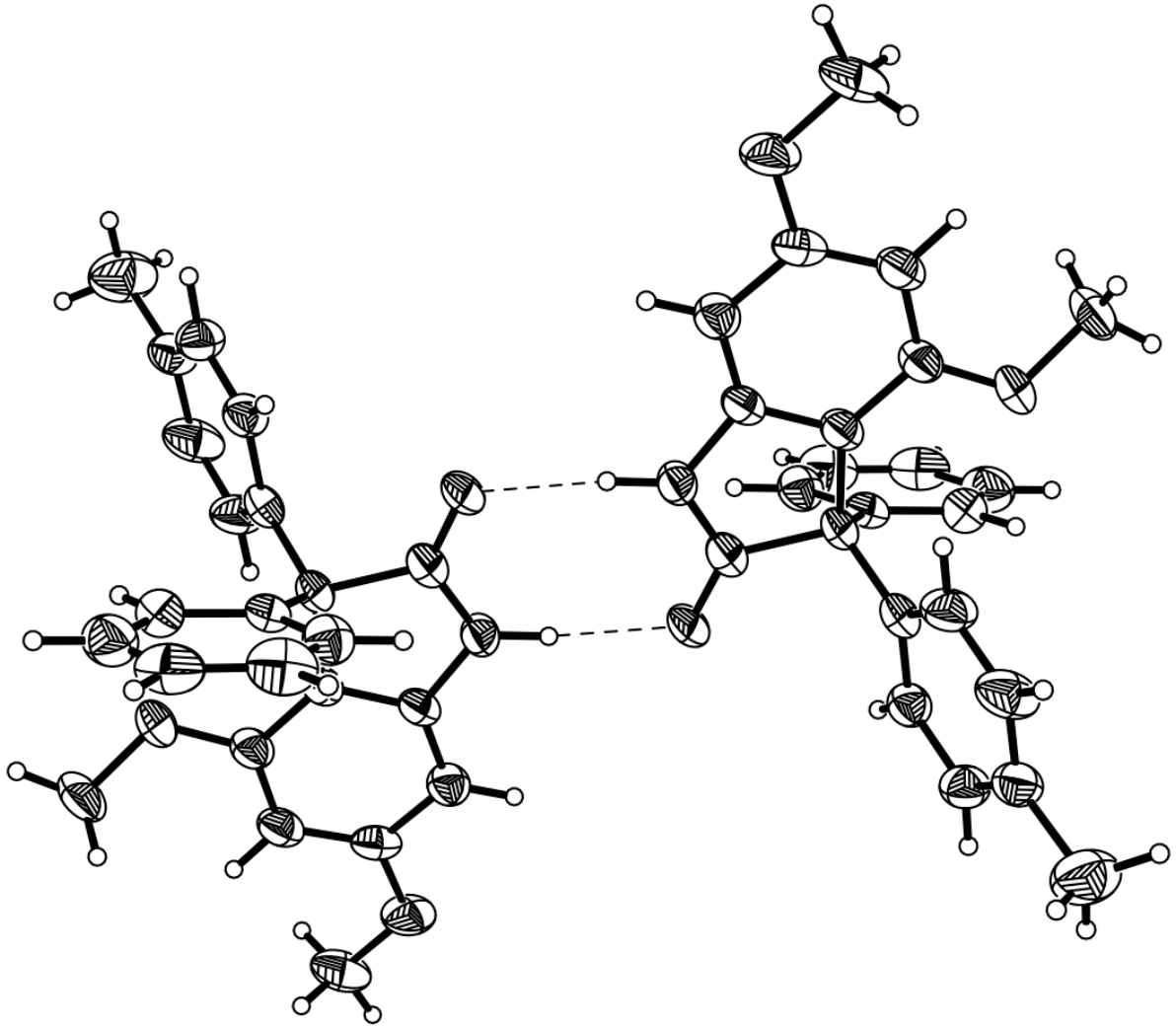


Figure S65. ORTEP diagram of (+/-)-**7** showing intermolecular hydrogen bonding with thermal ellipsoids drawn at 50% probability level.

Experimental Details for the Structure Determination of (+/-)-**7**.

Data collection

A crystal (0.26 x 0.12 x 0.08 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 173.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 90 seconds and a detector distance of 3.98 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 1741 strong reflections from the actual data collection after integration.[3] See Table S55 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group *P*-1 was determined based on intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. The amine hydrogen atoms were found from the difference Fourier map, and their positional and isotropic displacement parameters were refined independently from those of their bonded nitrogen atoms. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0663$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1646$ (F^2, all data).

Structure description

The structure is the one suggested. There are two independent molecules in the asymmetric unit with all atoms in general positions. Moderate intermolecular hydrogen bonding is observed: each molecule is hydrogen-bonded to its inverted symmetry equivalent (see Figure S65 and Table S61).

References

[1] *APEX2*, version 2011.4-1; Bruker AXS: Madison, WI, 2011.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.68A; Bruker AXS: Madison, WI, 2009.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| / \Sigma|F_o^2|$

$R1 = \Sigma||F_o| - |F_c|| / \Sigma|F_o|$

$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$

where $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$P = 1/3 \max(0, F_o^2) + 2/3\ F_c^2$

$GooF = S = [\Sigma[w(F_o^2 - F_c^2)^2] / (m-n)]^{1/2}$

where m = number of reflections and n = number of parameters

Table S55. Crystal data and structure refinement for (+/-)-**7**.

Identification code	(+/-)-**7**, JONJK61	
Empirical formula	C23 H21 N O3	
Formula weight	359.41	
Temperature	173.0(5) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 8.2312(16) Å	α = 81.845(4)°
	b = 9.912(2) Å	β = 87.012(4)°
	c = 23.000(5) Å	γ = 89.587(3)°
Volume	1854.9(6) Å^3	
Z	4	
Density (calculated)	1.287 Mg/m^3	
Absorption coefficient	0.085 mm^{-1}	
F(000)	760	
Crystal color, morphology	colorless, block	
Crystal size	0.26 x 0.12 x 0.08 mm^3	
Theta range for data collection	1.79 to 25.03°	
Index ranges	$-9 \le h \le 9$, $-11 \le k \le 11$, $-27 \le l \le 27$	
Reflections collected	16814	
Independent reflections	6550 [R(int) = 0.1103]	
Observed reflections	2958	
Completeness to theta = 25.03°	99.8%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9932 and 0.9782	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6550 / 0 / 502	
Goodness-of-fit on F^2	0.961	
Final R indices [I>2sigma(I)]	R1 = 0.0663, wR2 = 0.1259	
R indices (all data)	R1 = 0.1722, wR2 = 0.1646	
Extinction coefficient	0.0050(8)	
Largest diff. peak and hole	0.306 and -0.230 e.Å^{-3}	

Table S56. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for (+/-)-**7**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	9197(3)	9594(3)	3540(1)	49(1)
O2	7345(4)	10864(3)	5424(1)	49(1)
O3	5793(3)	4894(2)	4245(1)	41(1)
N1	5923(4)	6636(3)	4790(2)	35(1)
C1	6268(5)	6018(4)	4312(2)	36(1)
C2	7370(5)	6966(4)	3870(2)	34(1)
C3	7546(5)	8167(4)	4206(2)	34(1)
C4	6689(5)	7911(4)	4744(2)	32(1)
C7	9007(5)	6274(4)	3783(2)	35(1)
C8	6490(5)	7303(4)	3297(2)	34(1)
C9	8396(5)	9383(4)	4077(2)	38(1)
C10	8367(5)	10296(4)	4480(2)	40(1)
C11	7473(5)	10001(4)	5003(2)	38(1)
C12	6620(5)	8783(4)	5154(2)	38(1)
C13	10465(5)	10586(4)	3439(2)	56(1)
C14	8082(5)	12174(4)	5286(2)	54(1)
C15	9556(5)	5217(4)	4185(2)	42(1)
C16	11066(6)	4658(5)	4113(2)	58(1)
C17	12109(6)	5137(5)	3644(2)	55(1)
C18	11581(6)	6185(5)	3238(2)	55(1)
C19	10058(5)	6744(4)	3303(2)	45(1)
C20	6352(5)	6324(4)	2927(2)	37(1)
C21	5565(5)	6612(4)	2407(2)	42(1)
C22	4918(5)	7887(4)	2234(2)	43(1)
C23	5024(5)	8840(4)	2606(2)	54(1)
C24	5789(5)	8573(4)	3128(2)	48(1)
C25	4118(6)	8214(5)	1655(2)	73(2)
O4	9618(3)	8865(3)	8351(1)	46(1)
O5	7598(3)	10974(3)	10027(1)	48(1)
O6	5699(3)	4594(2)	9275(1)	40(1)
N2	5844(4)	6578(3)	9674(2)	36(1)
C31	6200(5)	5765(4)	9264(2)	33(1)
C32	7324(5)	6539(4)	8767(2)	33(1)

C33	7642(5)	7834(4)	9026(2)	33(1)
C34	6713(5)	7818(4)	9546(2)	35(1)
C37	8813(5)	5637(4)	8719(2)	33(1)
C38	6341(5)	6838(4)	8217(2)	36(1)
C39	8675(5)	8914(4)	8851(2)	35(1)
C40	8713(5)	9984(4)	9187(2)	38(1)
C41	7691(5)	9952(4)	9688(2)	39(1)
C42	6689(5)	8849(4)	9883(2)	38(1)
C43	10856(6)	9862(4)	8202(2)	70(2)
C44	8500(5)	12187(4)	9832(2)	50(1)
C45	9971(5)	5597(4)	9132(2)	46(1)
C46	11268(5)	4728(5)	9136(2)	50(1)
C47	11477(6)	3867(5)	8717(2)	51(1)
C48	10337(6)	3898(4)	8305(2)	54(1)
C49	9015(5)	4759(4)	8302(2)	43(1)
C50	7107(5)	7215(4)	7663(2)	42(1)
C51	6213(6)	7480(4)	7172(2)	45(1)
C52	4545(6)	7388(4)	7198(2)	42(1)
C53	3778(5)	7046(4)	7747(2)	47(1)
C54	4663(5)	6781(4)	8244(2)	45(1)
C55	3598(6)	7620(5)	6664(2)	65(2)

Table S57. Bond lengths [Å] and angles [°] for (+/-)-**7**.

O(1)-C(9)	1.360(5)	C(17)-H(17A)	0.9500
O(1)-C(13)	1.426(4)	C(18)-C(19)	1.376(6)
O(2)-C(11)	1.378(5)	C(18)-H(18A)	0.9500
O(2)-C(14)	1.425(4)	C(19)-H(19B)	0.9500
O(3)-C(1)	1.216(4)	C(20)-C(21)	1.383(5)
N(1)-C(1)	1.349(5)	C(20)-H(20A)	0.9500
N(1)-C(4)	1.404(4)	C(21)-C(22)	1.382(5)
N(1)-H(1)	0.94(4)	C(21)-H(21A)	0.9500
C(1)-C(2)	1.544(5)	C(22)-C(23)	1.369(6)
C(2)-C(3)	1.521(5)	C(22)-C(25)	1.509(6)
C(2)-C(7)	1.524(5)	C(23)-C(24)	1.376(6)
C(2)-C(8)	1.532(5)	C(23)-H(23B)	0.9500
C(3)-C(4)	1.382(5)	C(24)-H(24B)	0.9500
C(3)-C(9)	1.385(5)	C(25)-H(25A)	0.9800
C(4)-C(12)	1.366(5)	C(25)-H(25B)	0.9800
C(7)-C(15)	1.386(5)	C(25)-H(25C)	0.9800
C(7)-C(19)	1.394(5)	O(4)-C(39)	1.360(4)
C(8)-C(20)	1.386(5)	O(4)-C(43)	1.420(4)
C(8)-C(24)	1.394(5)	O(5)-C(41)	1.364(4)
C(9)-C(10)	1.383(5)	O(5)-C(44)	1.423(4)
C(10)-C(11)	1.373(5)	O(6)-C(31)	1.231(4)
C(10)-H(10A)	0.9500	N(2)-C(31)	1.346(5)
C(11)-C(12)	1.393(5)	N(2)-C(34)	1.413(5)
C(12)-H(12A)	0.9500	N(2)-H(2)	0.93(4)
C(13)-H(13A)	0.9800	C(31)-C(32)	1.549(5)
C(13)-H(13B)	0.9800	C(32)-C(33)	1.519(5)
C(13)-H(13C)	0.9800	C(32)-C(37)	1.521(5)
C(14)-H(14A)	0.9800	C(32)-C(38)	1.530(5)
C(14)-H(14B)	0.9800	C(33)-C(39)	1.373(5)
C(14)-H(14C)	0.9800	C(33)-C(34)	1.384(5)
C(15)-C(16)	1.369(6)	C(34)-C(42)	1.368(5)
C(15)-H(15A)	0.9500	C(37)-C(45)	1.376(5)
C(16)-C(17)	1.374(6)	C(37)-C(49)	1.387(5)
C(16)-H(16A)	0.9500	C(38)-C(54)	1.380(5)
C(17)-C(18)	1.379(6)	C(38)-C(50)	1.393(5)

C(39)-C(40)	1.400(5)	O(3)-C(1)-N(1)	125.2(4)
C(40)-C(41)	1.386(5)	O(3)-C(1)-C(2)	126.0(4)
C(40)-H(40A)	0.9500	N(1)-C(1)-C(2)	108.8(3)
C(41)-C(42)	1.383(5)	C(3)-C(2)-C(7)	110.8(3)
C(42)-H(42A)	0.9500	C(3)-C(2)-C(8)	114.3(3)
C(43)-H(43A)	0.9800	C(7)-C(2)-C(8)	112.7(3)
C(43)-H(43B)	0.9800	C(3)-C(2)-C(1)	100.6(3)
C(43)-H(43C)	0.9800	C(7)-C(2)-C(1)	109.3(3)
C(44)-H(44A)	0.9800	C(8)-C(2)-C(1)	108.4(3)
C(44)-H(44B)	0.9800	C(4)-C(3)-C(9)	117.9(4)
C(44)-H(44C)	0.9800	C(4)-C(3)-C(2)	109.7(3)
C(45)-C(46)	1.366(6)	C(9)-C(3)-C(2)	132.4(4)
C(45)-H(45A)	0.9500	C(12)-C(4)-C(3)	124.2(3)
C(46)-C(47)	1.377(6)	C(12)-C(4)-N(1)	126.7(4)
C(46)-H(46A)	0.9500	C(3)-C(4)-N(1)	109.1(3)
C(47)-C(48)	1.367(6)	C(15)-C(7)-C(19)	117.2(4)
C(47)-H(47A)	0.9500	C(15)-C(7)-C(2)	122.4(4)
C(48)-C(49)	1.378(6)	C(19)-C(7)-C(2)	120.4(4)
C(48)-H(48A)	0.9500	C(20)-C(8)-C(24)	117.6(4)
C(49)-H(49A)	0.9500	C(20)-C(8)-C(2)	120.1(3)
C(50)-C(51)	1.373(5)	C(24)-C(8)-C(2)	122.4(3)
C(50)-H(50A)	0.9500	O(1)-C(9)-C(10)	124.5(3)
C(51)-C(52)	1.374(6)	O(1)-C(9)-C(3)	115.6(4)
C(51)-H(51A)	0.9500	C(10)-C(9)-C(3)	119.9(4)
C(52)-C(53)	1.382(6)	C(11)-C(10)-C(9)	119.8(4)
C(52)-C(55)	1.478(6)	C(11)-C(10)-H(10A)	120.1
C(53)-C(54)	1.380(6)	C(9)-C(10)-H(10A)	120.1
C(53)-H(53A)	0.9500	C(10)-C(11)-O(2)	123.7(3)
C(54)-H(54A)	0.9500	C(10)-C(11)-C(12)	122.1(4)
C(55)-H(55A)	0.9800	O(2)-C(11)-C(12)	114.2(4)
C(55)-H(55B)	0.9800	C(4)-C(12)-C(11)	116.0(4)
C(55)-H(55C)	0.9800	C(4)-C(12)-H(12A)	122.0
C(9)-O(1)-C(13)	118.7(3)	C(11)-C(12)-H(12A)	122.0
C(11)-O(2)-C(14)	117.8(3)	O(1)-C(13)-H(13A)	109.5
C(1)-N(1)-C(4)	111.8(3)	O(1)-C(13)-H(13B)	109.5
C(1)-N(1)-H(1)	127(2)	H(13A)-C(13)-H(13B)	109.5
C(4)-N(1)-H(1)	121(2)	O(1)-C(13)-H(13C)	109.5

H(13A)-C(13)-H(13C)	109.5	C(8)-C(24)-H(24B)	119.7
H(13B)-C(13)-H(13C)	109.5	C(22)-C(25)-H(25A)	109.5
O(2)-C(14)-H(14A)	109.5	C(22)-C(25)-H(25B)	109.5
O(2)-C(14)-H(14B)	109.5	H(25A)-C(25)-H(25B)	109.5
H(14A)-C(14)-H(14B)	109.5	C(22)-C(25)-H(25C)	109.5
O(2)-C(14)-H(14C)	109.5	H(25A)-C(25)-H(25C)	109.5
H(14A)-C(14)-H(14C)	109.5	H(25B)-C(25)-H(25C)	109.5
H(14B)-C(14)-H(14C)	109.5	C(39)-O(4)-C(43)	118.1(3)
C(16)-C(15)-C(7)	121.3(4)	C(41)-O(5)-C(44)	118.2(3)
C(16)-C(15)-H(15A)	119.4	C(31)-N(2)-C(34)	110.7(4)
C(7)-C(15)-H(15A)	119.4	C(31)-N(2)-H(2)	128(2)
C(15)-C(16)-C(17)	121.5(5)	C(34)-N(2)-H(2)	121(2)
C(15)-C(16)-H(16A)	119.3	O(6)-C(31)-N(2)	125.6(4)
C(17)-C(16)-H(16A)	119.3	O(6)-C(31)-C(32)	125.0(4)
C(16)-C(17)-C(18)	118.0(5)	N(2)-C(31)-C(32)	109.4(3)
C(16)-C(17)-H(17A)	121.0	C(33)-C(32)-C(37)	114.0(3)
C(18)-C(17)-H(17A)	121.0	C(33)-C(32)-C(38)	111.1(3)
C(19)-C(18)-C(17)	121.0(5)	C(37)-C(32)-C(38)	116.3(3)
C(19)-C(18)-H(18A)	119.5	C(33)-C(32)-C(31)	100.5(3)
C(17)-C(18)-H(18A)	119.5	C(37)-C(32)-C(31)	105.5(3)
C(18)-C(19)-C(7)	121.0(4)	C(38)-C(32)-C(31)	107.8(3)
C(18)-C(19)-H(19B)	119.5	C(39)-C(33)-C(34)	118.3(3)
C(7)-C(19)-H(19B)	119.5	C(39)-C(33)-C(32)	132.4(4)
C(21)-C(20)-C(8)	120.8(4)	C(34)-C(33)-C(32)	109.2(3)
C(21)-C(20)-H(20A)	119.6	C(42)-C(34)-C(33)	124.1(4)
C(8)-C(20)-H(20A)	119.6	C(42)-C(34)-N(2)	126.1(4)
C(22)-C(21)-C(20)	121.3(4)	C(33)-C(34)-N(2)	109.8(3)
C(22)-C(21)-H(21A)	119.3	C(45)-C(37)-C(49)	117.3(4)
C(20)-C(21)-H(21A)	119.3	C(45)-C(37)-C(32)	119.4(4)
C(23)-C(22)-C(21)	117.6(4)	C(49)-C(37)-C(32)	123.1(4)
C(23)-C(22)-C(25)	121.2(4)	C(54)-C(38)-C(50)	116.8(4)
C(21)-C(22)-C(25)	121.2(4)	C(54)-C(38)-C(32)	122.0(4)
C(22)-C(23)-C(24)	122.1(4)	C(50)-C(38)-C(32)	121.1(4)
C(22)-C(23)-H(23B)	119.0	O(4)-C(39)-C(33)	116.6(3)
C(24)-C(23)-H(23B)	119.0	O(4)-C(39)-C(40)	123.8(3)
C(23)-C(24)-C(8)	120.5(4)	C(33)-C(39)-C(40)	119.6(4)
C(23)-C(24)-H(24B)	119.7	C(41)-C(40)-C(39)	119.7(3)

C(41)-C(40)-H(40A)	120.1	C(46)-C(47)-H(47A)	121.1
C(39)-C(40)-H(40A)	120.1	C(47)-C(48)-C(49)	121.6(4)
O(5)-C(41)-C(42)	114.8(4)	C(47)-C(48)-H(48A)	119.2
O(5)-C(41)-C(40)	123.7(3)	C(49)-C(48)-H(48A)	119.2
C(42)-C(41)-C(40)	121.6(4)	C(48)-C(49)-C(37)	120.5(4)
C(34)-C(42)-C(41)	116.6(4)	C(48)-C(49)-H(49A)	119.7
C(34)-C(42)-H(42A)	121.7	C(37)-C(49)-H(49A)	119.7
C(41)-C(42)-H(42A)	121.7	C(51)-C(50)-C(38)	120.7(4)
O(4)-C(43)-H(43A)	109.5	C(51)-C(50)-H(50A)	119.7
O(4)-C(43)-H(43B)	109.5	C(38)-C(50)-H(50A)	119.7
H(43A)-C(43)-H(43B)	109.5	C(50)-C(51)-C(52)	122.5(4)
O(4)-C(43)-H(43C)	109.5	C(50)-C(51)-H(51A)	118.8
H(43A)-C(43)-H(43C)	109.5	C(52)-C(51)-H(51A)	118.8
H(43B)-C(43)-H(43C)	109.5	C(51)-C(52)-C(53)	117.1(4)
O(5)-C(44)-H(44A)	109.5	C(51)-C(52)-C(55)	122.1(4)
O(5)-C(44)-H(44B)	109.5	C(53)-C(52)-C(55)	120.8(4)
H(44A)-C(44)-H(44B)	109.5	C(54)-C(53)-C(52)	121.0(4)
O(5)-C(44)-H(44C)	109.5	C(54)-C(53)-H(53A)	119.5
H(44A)-C(44)-H(44C)	109.5	C(52)-C(53)-H(53A)	119.5
H(44B)-C(44)-H(44C)	109.5	C(53)-C(54)-C(38)	121.9(4)
C(46)-C(45)-C(37)	121.8(4)	C(53)-C(54)-H(54A)	119.0
C(46)-C(45)-H(45A)	119.1	C(38)-C(54)-H(54A)	119.0
C(37)-C(45)-H(45A)	119.1	C(52)-C(55)-H(55A)	109.5
C(45)-C(46)-C(47)	120.9(4)	C(52)-C(55)-H(55B)	109.5
C(45)-C(46)-H(46A)	119.6	H(55A)-C(55)-H(55B)	109.5
C(47)-C(46)-H(46A)	119.6	C(52)-C(55)-H(55C)	109.5
C(48)-C(47)-C(46)	117.9(4)	H(55A)-C(55)-H(55C)	109.5
C(48)-C(47)-H(47A)	121.1	H(55B)-C(55)-H(55C)	109.5

Table S58. Anisotropic displacement parameters ($Å^2$ x 10^3) for (+/-)-**7**. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [h^2 a^{*2} U_{11} + ... + 2 h k a* b* U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	58(2)	42(2)	46(2)	0(2)	4(2)	-26(2)
O2	67(2)	31(2)	54(2)	-15(2)	-14(2)	-6(2)
O3	51(2)	27(2)	45(2)	-6(1)	2(2)	-16(1)
N1	39(2)	30(2)	35(2)	-6(2)	6(2)	-6(2)
C1	33(3)	32(2)	42(3)	-2(2)	0(2)	-6(2)
C2	40(3)	27(2)	36(2)	-1(2)	1(2)	-11(2)
C3	33(2)	29(2)	38(3)	-3(2)	-4(2)	-5(2)
C4	31(2)	27(2)	38(3)	-4(2)	-5(2)	-5(2)
C7	36(3)	32(2)	37(3)	-9(2)	-3(2)	-8(2)
C8	34(3)	26(2)	40(3)	-1(2)	1(2)	-7(2)
C9	41(3)	30(2)	41(3)	-3(2)	-5(2)	-8(2)
C10	41(3)	32(2)	46(3)	-1(2)	-11(2)	-8(2)
C11	41(3)	30(2)	45(3)	-11(2)	-16(2)	-1(2)
C12	40(3)	34(2)	39(3)	-2(2)	-4(2)	-3(2)
C13	52(3)	50(3)	63(3)	5(2)	-3(3)	-27(2)
C14	58(3)	30(2)	79(4)	-19(2)	-17(3)	-7(2)
C15	48(3)	32(2)	47(3)	-4(2)	-4(2)	-1(2)
C16	59(4)	45(3)	73(4)	-14(3)	-17(3)	9(3)
C17	45(3)	57(3)	68(4)	-27(3)	-13(3)	7(3)
C18	42(3)	69(4)	58(3)	-23(3)	3(3)	-7(3)
C19	49(3)	47(3)	41(3)	-6(2)	-2(2)	-9(2)
C20	40(3)	32(2)	40(3)	-3(2)	-5(2)	-3(2)
C21	44(3)	42(3)	42(3)	-14(2)	-3(2)	-4(2)
C22	42(3)	40(3)	48(3)	-2(2)	-13(2)	-6(2)
C23	59(3)	30(3)	75(4)	-5(2)	-26(3)	-2(2)
C24	59(3)	34(3)	53(3)	-12(2)	-15(3)	-7(2)
C25	89(4)	61(3)	71(4)	-6(3)	-36(3)	0(3)
O4	56(2)	32(2)	49(2)	-7(1)	12(2)	-20(1)
O5	52(2)	32(2)	61(2)	-18(2)	1(2)	-9(1)
O6	46(2)	28(2)	47(2)	-8(1)	6(1)	-13(1)
N2	43(2)	28(2)	36(2)	-6(2)	7(2)	-11(2)
C31	34(3)	30(2)	37(3)	-6(2)	-5(2)	-6(2)
C32	38(3)	25(2)	36(2)	-4(2)	2(2)	-10(2)

C33	37(3)	24(2)	37(3)	-5(2)	0(2)	-4(2)
C34	35(3)	28(2)	42(3)	-3(2)	-4(2)	-5(2)
C37	36(3)	22(2)	40(3)	-4(2)	3(2)	-7(2)
C38	41(3)	24(2)	44(3)	-9(2)	-2(2)	-6(2)
C39	37(3)	31(2)	37(3)	-5(2)	-4(2)	-6(2)
C40	40(3)	27(2)	47(3)	-5(2)	-6(2)	-8(2)
C41	44(3)	26(2)	50(3)	-12(2)	-15(2)	0(2)
C42	39(3)	32(2)	43(3)	-9(2)	1(2)	-5(2)
C43	76(4)	44(3)	91(4)	-24(3)	38(3)	-34(3)
C44	60(3)	33(2)	59(3)	-10(2)	-11(3)	-7(2)
C45	47(3)	44(3)	49(3)	-15(2)	-11(2)	-6(2)
C46	35(3)	52(3)	61(3)	-3(3)	-10(2)	-3(2)
C47	49(3)	51(3)	51(3)	0(2)	5(3)	6(2)
C48	66(4)	47(3)	48(3)	-11(2)	7(3)	12(3)
C49	47(3)	40(3)	42(3)	-9(2)	-5(2)	-1(2)
C50	43(3)	41(3)	41(3)	-2(2)	-5(2)	-11(2)
C51	57(3)	38(3)	39(3)	3(2)	-2(2)	-11(2)
C52	52(3)	26(2)	49(3)	-7(2)	-15(3)	-3(2)
C53	40(3)	46(3)	58(3)	-10(2)	-8(3)	-1(2)
C54	42(3)	46(3)	48(3)	-9(2)	-3(2)	-7(2)
C55	68(4)	56(3)	70(4)	-2(3)	-19(3)	0(3)

Table S59. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for (+/-)-**7**.

	x	y	z	$U_{(eq)}$
H1	5240(40)	6310(40)	5120(16)	36(11)
H10A	8964	11123	4395	47
H12A	6025	8571	5520	46
H13A	11022	10540	3055	84
H13B	11246	10401	3748	84
H13C	10000	11497	3445	84
H14A	7859	12696	5613	81
H14B	7635	12659	4929	81
H14C	9260	12069	5223	81
H15A	8871	4872	4517	51
H16A	11400	3924	4393	70
H17A	13164	4756	3601	66
H18A	12278	6527	2909	66
H19B	9717	7461	3016	54
H20A	6804	5444	3033	45
H21A	5467	5919	2165	50
H23B	4555	9713	2501	65
H24B	5840	9261	3375	57
H25A	4646	9011	1422	109
H25B	2963	8410	1728	109
H25C	4226	7432	1438	109
H2	5220(40)	6380(40)	10026(16)	37(12)
H40A	9436	10728	9073	45
H42A	6017	8810	10234	45
H43A	11434	9710	7833	105
H43B	11624	9792	8516	105
H43C	10366	10772	8153	105
H44A	8215	12872	10089	75
H44B	8239	12538	9428	75
H44C	9666	11988	9846	75
H45A	9866	6190	9422	55
H46A	12035	4717	9431	59
H47A	12386	3270	8716	61

H48A	10458	3311	8012	64
H49A	8235	4751	8012	51
H50A	8259	7289	7626	50
H51A	6769	7736	6801	54
H53A	2625	6992	7783	57
H54A	4101	6553	8616	54
H55A	3870	8516	6443	98
H55B	2433	7586	6778	98
H55C	3862	6912	6417	98

Table S60. Torsion angles [°] for (+/-)-**7**.

C4-N1-C1-O3	178.2(4)	C2-C3-C9-O1	2.5(7)
C4-N1-C1-C2	-1.3(5)	C4-C3-C9-C10	0.0(6)
O3-C1-C2-C3	-178.9(4)	C2-C3-C9-C10	-178.4(4)
N1-C1-C2-C3	0.6(4)	O1-C9-C10-C11	177.8(4)
O3-C1-C2-C7	-62.3(5)	C3-C9-C10-C11	-1.1(6)
N1-C1-C2-C7	117.2(4)	C9-C10-C11-O2	-178.4(4)
O3-C1-C2-C8	60.9(5)	C9-C10-C11-C12	2.0(6)
N1-C1-C2-C8	-119.6(4)	C14-O2-C11-C10	4.9(6)
C7-C2-C3-C4	-115.2(4)	C14-O2-C11-C12	-175.5(3)
C8-C2-C3-C4	116.2(4)	C3-C4-C12-C11	0.5(6)
C1-C2-C3-C4	0.3(4)	N1-C4-C12-C11	-179.3(4)
C7-C2-C3-C9	63.3(6)	C10-C11-C12-C4	-1.6(6)
C8-C2-C3-C9	-65.3(6)	O2-C11-C12-C4	178.7(4)
C1-C2-C3-C9	178.8(4)	C19-C7-C15-C16	0.0(6)
C9-C3-C4-C12	0.3(6)	C2-C7-C15-C16	-176.6(4)
C2-C3-C4-C12	179.1(4)	C7-C15-C16-C17	1.0(7)
C9-C3-C4-N1	-179.8(4)	C15-C16-C17-C18	-1.3(7)
C2-C3-C4-N1	-1.1(5)	C16-C17-C18-C19	0.5(6)
C1-N1-C4-C12	-178.6(4)	C17-C18-C19-C7	0.5(6)
C1-N1-C4-C3	1.5(5)	C15-C7-C19-C18	-0.7(6)
C3-C2-C7-C15	90.0(4)	C2-C7-C19-C18	175.9(4)
C8-C2-C7-C15	-140.6(4)	C24-C8-C20-C21	0.9(6)
C1-C2-C7-C15	-20.0(5)	C2-C8-C20-C21	-179.8(4)
C3-C2-C7-C19	-86.5(4)	C8-C20-C21-C22	1.2(6)
C8-C2-C7-C19	43.0(4)	C20-C21-C22-C23	-2.6(6)
C1-C2-C7-C19	163.5(3)	C20-C21-C22-C25	177.6(4)
C3-C2-C8-C20	175.4(3)	C21-C22-C23-C24	2.1(7)
C7-C2-C8-C20	47.8(5)	C25-C22-C23-C24	-178.1(4)
C1-C2-C8-C20	-73.3(5)	C22-C23-C24-C8	-0.1(7)
C3-C2-C8-C24	-5.3(5)	C20-C8-C24-C23	-1.4(6)
C7-C2-C8-C24	-132.9(4)	C2-C8-C24-C23	179.3(4)
C1-C2-C8-C24	106.0(4)	C34-N2-C31-O6	175.8(4)
C13-O1-C9-C10	19.5(6)	C34-N2-C31-C32	-5.0(4)
C13-O1-C9-C3	-161.5(4)	O6-C31-C32-C33	-174.7(4)
C4-C3-C9-O1	-179.1(4)	N2-C31-C32-C33	6.1(4)

O6-C31-C32-C37	-55.9(5)	C32-C33-C39-O4	0.3(7)
N2-C31-C32-C37	124.9(3)	C34-C33-C39-C40	-2.5(6)
O6-C31-C32-C38	68.9(5)	C32-C33-C39-C40	-179.5(4)
N2-C31-C32-C38	-110.2(4)	O4-C39-C40-C41	179.2(4)
C37-C32-C33-C39	59.7(6)	C33-C39-C40-C41	-1.0(6)
C38-C32-C33-C39	-74.1(6)	C44-O5-C41-C42	-175.4(4)
C31-C32-C33-C39	172.0(4)	C44-O5-C41-C40	5.3(6)
C37-C32-C33-C34	-117.5(4)	C39-C40-C41-O5	-177.2(4)
C38-C32-C33-C34	108.7(4)	C39-C40-C41-C42	3.5(6)
C31-C32-C33-C34	-5.2(4)	C33-C34-C42-C41	-1.4(6)
C39-C33-C34-C42	3.8(6)	N2-C34-C42-C41	177.2(4)
C32-C33-C34-C42	-178.5(4)	O5-C41-C42-C34	178.3(3)
C39-C33-C34-N2	-174.9(4)	C40-C41-C42-C34	-2.4(6)
C32-C33-C34-N2	2.7(5)	C49-C37-C45-C46	-0.3(6)
C31-N2-C34-C42	-177.2(4)	C32-C37-C45-C46	174.9(4)
C31-N2-C34-C33	1.5(5)	C37-C45-C46-C47	1.0(7)
C33-C32-C37-C45	32.5(5)	C45-C46-C47-C48	-0.9(6)
C38-C32-C37-C45	163.8(3)	C46-C47-C48-C49	0.1(7)
C31-C32-C37-C45	-76.8(4)	C47-C48-C49-C37	0.7(7)
C33-C32-C37-C49	-152.6(4)	C45-C37-C49-C48	-0.5(6)
C38-C32-C37-C49	-21.3(5)	C32-C37-C49-C48	-175.5(4)
C31-C32-C37-C49	98.1(4)	C54-C38-C50-C51	-1.6(6)
C33-C32-C38-C54	-91.5(4)	C32-C38-C50-C51	179.9(4)
C37-C32-C38-C54	135.8(4)	C38-C50-C51-C52	-0.1(6)
C31-C32-C38-C54	17.6(5)	C50-C51-C52-C53	1.6(6)
C33-C32-C38-C50	86.9(4)	C50-C51-C52-C55	-177.5(4)
C37-C32-C38-C50	-45.8(5)	C51-C52-C53-C54	-1.4(6)
C31-C32-C38-C50	-163.9(3)	C55-C52-C53-C54	177.7(4)
C43-O4-C39-C33	-171.4(4)	C52-C53-C54-C38	-0.3(6)
C43-O4-C39-C40	8.4(6)	C50-C38-C54-C53	1.8(6)
C34-C33-C39-O4	177.3(4)	C32-C38-C54-C53	-179.7(4)

Table S61. Hydrogen bonds and close contacts for (+/-)-**7** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1...O3#1	0.94(4)	1.92(4)	2.824(4)	162(3)
N2-H2...O6#2	0.93(4)	1.88(4)	2.776(4)	161(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1 #2 -x+1,-y+1,-z+2

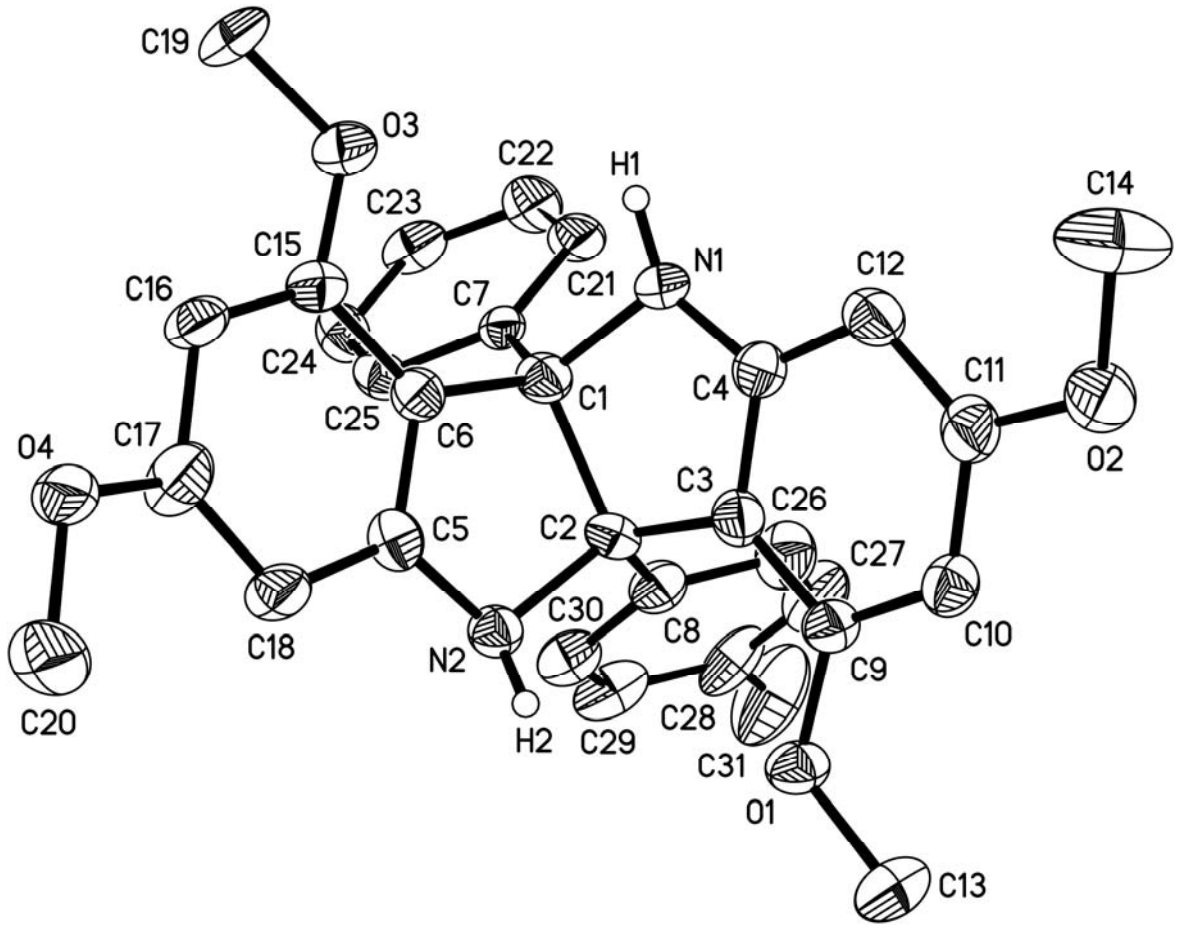


Figure S66. ORTEP diagram of **8** with thermal ellipsoids drawn at 50% probability level. Hydrogen atoms were omitted for clarity, except for H1 and H2, to emphasize valency of the nitrogen atoms.

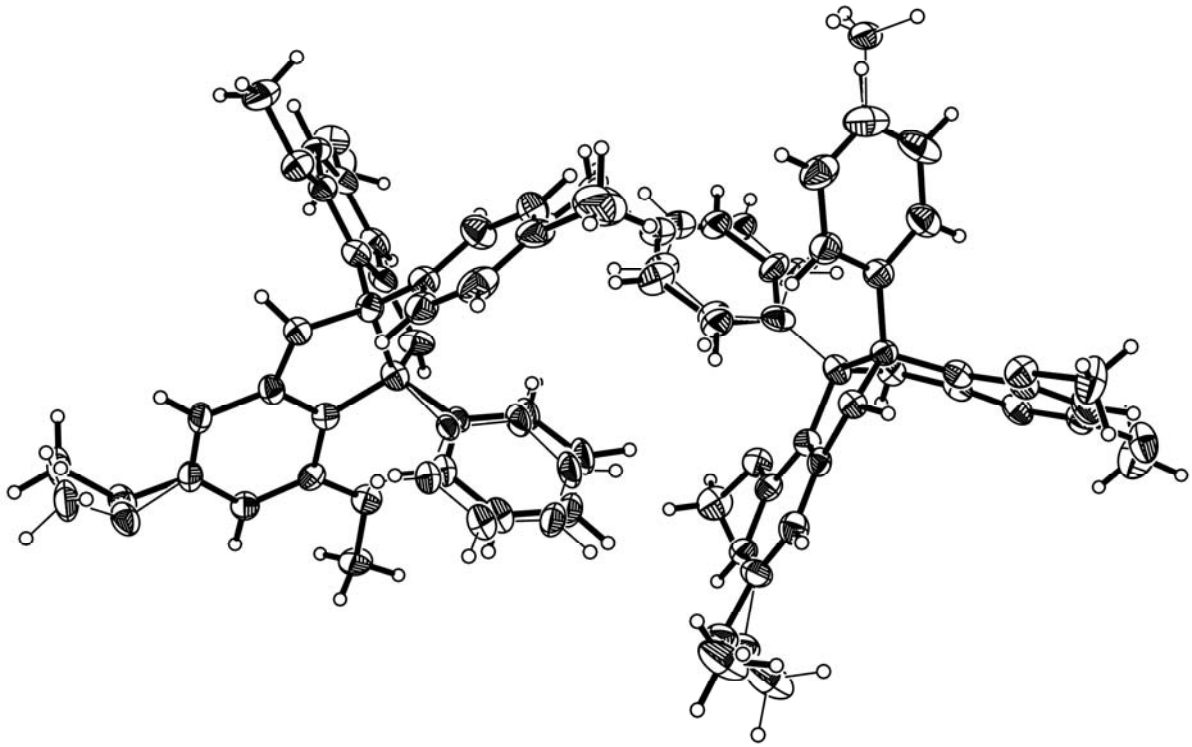


Figure S67. ORTEP diagram of asymmetric unit of **8** (disordered) with thermal ellipsoids drawn at 50% probability level.

Experimental Details for the Structure Determination of (+/-)-**8**.

Data collection

A crystal (0.40 x 0.16 x 0.08 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube or fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 3.98 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4058 strong reflections from the actual data collection after integration.[3] See Table S62 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR97[4] and refined using SHELXL-97.[5] The space group $P2_1$ was determined based on observed disorder ratios: due to the refined occupancies of the components of the disordered groups, the refinement had to be performed in chiral space group $P2_1$, despite significant pseudosymmetric effects and worse overall statistics. A refinement in centrosymmetric space group $P2_1/c$ requires that all disordered component ratios be 1:1 because of crystallographic symmetry (see below). However, this was clearly not the case, and forcing the ratios to be 1:1 resulted in an inaccurate and unrealistic model. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. Amine hydrogen atoms were found from the difference Fourier map, and their positional parameters were refined independently from those of their respective bonded nitrogen atoms. Their isotropic displacement parameters were relative to those of the nitrogen atoms, such that U_{iso}[H] = 1.5*U_{eq}[N]. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. Due to the lack of significant anomalous scattering effects, all Friedel opposites were merged in the final refinement. The final full matrix least squares refinement converged to $R1$ = 0.0559 (F^2, $I > 2\sigma(I)$) and $wR2$ = 0.1372 (F^2, all data).

Structure description

The structure is the one suggested. The structure was modeled in space group $P2_1$ with two independent molecules and all atoms in general positions. Phenyl group C7, C21-C25 on one independent molecule and methoxy group O8-C70 on the other independent molecule are modeled as disordered over two positions each (75:25). Initially the ratios of each were determined independently, which resulted in a common ratio. Further examination showed that the disorders had to be refined together to avoid any violation of van der Waals radii. Phenyl group C57, C71-C75 on one independent molecule and methoxy group O4-C20 on the other independent molecule are modeled as disordered over two positions (50:50 exactly, a requirement of the crystallographic two-fold screw axes). Independent refinements of these disorders also resulted in the 50:50 ratio. Additionally, methyl group C81 on one molecule is disordered (50:50, again a requirement of symmetry) over the two possible phenyl ring locations, while methyl group C31 only exists on one phenyl ring in the other molecule. Weak to moderate intermolecular and weak intramolecular hydrogen bonding is observed (see

Table S68).

References

[1] *APEX2*, version 2011.4-1; Bruker AXS: Madison, WI, 2011.

[2] Sheldrick, G. M. *SADABS*, version 2008/1; University of Göttingen: Göttingen, Germany, 2008.

[3] *SAINT*, version 7.68A; Bruker AXS: Madison, WI, 2009.

[4] Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *SIR97: A new program for solving and refining crystal structures*; Istituto di Cristallografia, CNR: Bari, Italy, 1999.

[5] Sheldrick, G. M. *Acta. Cryst.* **2008**, *A64*, 112-122.

Some equations of interest:

$$R_{int} = \Sigma|F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma|F_o^2|$$

$$R1 = \Sigma||F_o| - |F_c|| \,/\, \Sigma|F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma[w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2(F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max(0, F_o^2) + 2/3\, F_c^2$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

Table S62. Crystal data and structure refinement for (+/-)-**8**.

Identification code	(+/-)-**8**, JONJK63	
Empirical formula	C31 H30 N2 O4	
Formula weight	494.57	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	a = 11.6386(8) Å	α = 90°
	b = 19.1068(13) Å	β = 94.728(1)°
	c = 11.7615(8) Å	γ = 90°
Volume	2606.6(3) Å^3	
Z	4	
Density (calculated)	1.260 Mg/m^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	1048	
Crystal color, morphology	colorless, needle	
Crystal size	0.40 x 0.16 x 0.08 mm^3	
Theta range for data collection	1.74 to 28.28°	
Index ranges	$-15 \le h \le 15$, $-25 \le k \le 25$, $-15 \le l \le 15$	
Reflections collected	37187	
Independent reflections	6662 [R(int) = 0.0869]	
Observed reflections	4153	
Completeness to theta = 28.28°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9933 and 0.9673	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6662 / 30 / 657	
Goodness-of-fit on F^2	1.022	
Final R indices [I>2sigma(I)]	R1 = 0.0559, wR2 = 0.1155	
R indices (all data)	R1 = 0.1029, wR2 = 0.1372	
Largest diff. peak and hole	0.346 and -0.362 e.Å^{-3}	

Table S63. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for (+/-)-**8**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N1	4948(4)	-551(3)	1426(4)	33(1)
N2	7090(4)	-886(3)	3667(4)	26(1)
C1	5945(4)	-267(3)	2137(5)	27(1)
C2	5870(4)	-666(3)	3340(5)	28(1)
C3	5132(4)	-1293(3)	2958(5)	28(1)
C4	4658(4)	-1200(4)	1855(5)	29(1)
C5	7677(4)	-890(3)	2649(5)	27(1)
C6	7082(4)	-527(3)	1790(5)	25(1)
C9	4904(5)	-1893(3)	3553(5)	28(1)
C10	4215(4)	-2412(3)	3024(5)	29(1)
C11	3774(5)	-2321(4)	1899(5)	34(2)
C12	3975(4)	-1717(4)	1287(5)	32(1)
O1	5418(3)	-1914(2)	4646(3)	30(1)
C13	5208(6)	-2524(4)	5308(6)	42(2)
O2	3125(4)	-2871(3)	1476(4)	40(1)
C14	2882(6)	-2925(4)	285(5)	60(1)
C15	7595(4)	-409(3)	773(5)	26(1)
C16	8643(5)	-699(3)	613(5)	30(1)
C17	9199(5)	-1084(4)	1515(7)	43(2)
C18	8753(5)	-1196(3)	2540(5)	33(1)
O3	6967(3)	-21(2)	-19(3)	31(1)
C19	7521(5)	161(4)	-1008(5)	38(2)
O4	10098(6)	-1482(5)	1097(8)	36(2)
C20	10628(10)	-1979(7)	1883(11)	45(2)
O4'	10374(6)	-1243(6)	1383(9)	36(2)
C20'	11092(10)	-1531(8)	2328(12)	45(2)
C7	5848(10)	532(4)	2190(20)	22(2)
C21	4862(10)	893(7)	1824(18)	36(2)
C22	4850(10)	1617(6)	1899(14)	40(2)
C23	5844(10)	1992(6)	2257(14)	36(3)
C24	6813(10)	1615(6)	2618(15)	35(2)
C25	6829(10)	906(6)	2592(17)	27(2)
C7'	6090(30)	531(9)	2240(60)	22(2)

C21'	5120(30)	915(18)	1980(50)	36(2)
C22'	5200(20)	1640(18)	1970(40)	40(2)
C23'	6210(30)	1972(16)	2400(40)	36(3)
C24'	7140(30)	1572(17)	2750(50)	35(2)
C25'	7110(30)	862(17)	2650(50)	27(2)
C8	5397(5)	-243(3)	4281(5)	30(1)
C26	4222(5)	-221(4)	4364(6)	41(1)
C27	3797(6)	198(4)	5219(7)	47(2)
C28	4485(6)	587(4)	5967(6)	44(2)
C29	5661(7)	557(4)	5860(6)	46(1)
C30	6131(5)	145(4)	5045(6)	36(1)
C31	3961(8)	1032(5)	6862(8)	84(3)
N3	11(4)	3120(3)	1415(5)	30(1)
N4	2164(4)	3451(3)	3655(4)	26(1)
C51	1010(4)	2841(3)	2146(5)	27(1)
C52	951(4)	3258(3)	3337(5)	24(1)
C53	216(4)	3862(3)	2948(5)	28(1)
C54	-252(4)	3783(4)	1831(5)	29(1)
C55	2766(4)	3462(3)	2694(5)	24(1)
C56	2161(4)	3095(3)	1805(5)	25(1)
C59	1(4)	4473(4)	3536(5)	28(1)
C60	-671(4)	4994(4)	3017(5)	34(2)
C61	-1126(4)	4893(4)	1878(5)	31(1)
C62	-934(4)	4294(4)	1279(5)	32(1)
O5	525(3)	4526(2)	4612(3)	30(1)
C63	316(6)	5132(4)	5247(6)	44(2)
O6	-1764(3)	5456(3)	1436(4)	42(1)
C64	-2240(6)	5373(4)	259(5)	60(1)
C65	2679(4)	2974(3)	800(5)	26(1)
C66	3774(4)	3235(3)	685(5)	30(1)
C67	4333(5)	3615(4)	1554(6)	34(1)
C68	3856(5)	3724(3)	2592(5)	33(1)
O7	2052(3)	2572(2)	-4(3)	31(1)
C69	2608(6)	2385(4)	-1015(6)	43(2)
O8	5471(6)	3791(5)	1393(9)	39(2)
C70	6146(11)	4090(9)	2336(12)	67(4)
O8'	5280(16)	3982(13)	1220(20)	39(2)

C70'	5760(30)	4480(20)	2020(30)	67(4)
C57	988(5)	2056(4)	2233(5)	32(1)
C71	112(18)	1573(7)	1900(40)	35(3)
C72	239(12)	849(7)	2066(14)	40(2)
C73	1255(12)	578(7)	2507(14)	33(2)
C74	2143(15)	1019(7)	2882(16)	35(3)
C75	1991(16)	1747(7)	2700(30)	40(2)
C57'	988(5)	2056(4)	2233(5)	32(1)
C71'	-54(19)	1735(7)	1850(50)	35(3)
C72'	-146(12)	1003(7)	1835(16)	40(2)
C73'	769(12)	611(7)	2237(14)	33(2)
C74'	1810(14)	920(7)	2624(17)	35(3)
C75'	1895(16)	1659(7)	2660(30)	40(2)
C58	462(5)	2822(3)	4263(5)	30(1)
C76	-722(5)	2771(4)	4345(6)	41(1)
C77	-1214(6)	2363(4)	5128(6)	49(2)
C78	-507(7)	1972(4)	5890(6)	51(2)
C79	672(7)	2012(4)	5855(6)	46(1)
C80	1143(5)	2429(3)	5035(6)	36(1)
C81	1438(8)	-219(4)	2599(8)	41(2)
C81'	-1135(12)	1534(7)	6734(12)	47(3)

Table S64. Bond lengths [Å] and angles [°] for (+/-)-**8**.

N(1)-C(4)	1.391(9)	C(16)-C(17)	1.404(9)
N(1)-C(1)	1.476(7)	C(16)-H(16)	0.9500
N(1)-H(1)	0.84(6)	C(17)-C(18)	1.369(9)
N(2)-C(5)	1.426(7)	C(17)-O(4)	1.414(9)
N(2)-C(2)	1.501(7)	C(17)-O(4')	1.421(9)
N(2)-H(2)	0.90(6)	C(18)-H(18)	0.9500
C(1)-C(6)	1.501(7)	O(3)-C(19)	1.419(7)
C(1)-C(7)	1.534(10)	C(19)-H(19A)	0.9800
C(1)-C(7')	1.539(17)	C(19)-H(19B)	0.9800
C(1)-C(2)	1.616(8)	C(19)-H(19C)	0.9800
C(2)-C(8)	1.510(8)	O(4)-C(20)	1.429(11)
C(2)-C(3)	1.520(9)	C(20)-H(20A)	0.9800
C(3)-C(4)	1.378(8)	C(20)-H(20B)	0.9800
C(3)-C(9)	1.381(8)	C(20)-H(20C)	0.9800
C(4)-C(12)	1.401(9)	O(4')-C(20')	1.443(11)
C(5)-C(6)	1.366(8)	C(20')-H(20D)	0.9800
C(5)-C(18)	1.398(7)	C(20')-H(20E)	0.9800
C(6)-C(15)	1.399(8)	C(20')-H(20F)	0.9800
C(9)-O(1)	1.373(7)	C(7)-C(21)	1.378(10)
C(9)-C(10)	1.390(8)	C(7)-C(25)	1.395(10)
C(10)-C(11)	1.390(8)	C(21)-C(22)	1.386(12)
C(10)-H(10)	0.9500	C(21)-H(21)	0.9500
C(11)-O(2)	1.364(8)	C(22)-C(23)	1.396(12)
C(11)-C(12)	1.391(9)	C(22)-H(22)	0.9500
C(12)-H(12)	0.9500	C(23)-C(24)	1.376(10)
O(1)-C(13)	1.433(8)	C(23)-H(23)	0.9500
C(13)-H(13A)	0.9800	C(24)-C(25)	1.355(11)
C(13)-H(13B)	0.9800	C(24)-H(24)	0.9500
C(13)-H(13C)	0.9800	C(25)-H(25)	0.9500
O(2)-C(14)	1.410(7)	C(7')-C(21')	1.365(18)
C(14)-H(14A)	0.9800	C(7')-C(25')	1.395(17)
C(14)-H(14B)	0.9800	C(21')-C(22')	1.389(19)
C(14)-H(14C)	0.9800	C(21')-H(21')	0.9500
C(15)-O(3)	1.355(7)	C(22')-C(23')	1.388(19)
C(15)-C(16)	1.366(7)	C(22')-H(22')	0.9500

C(23')-C(24')	1.360(19)	C(59)-C(60)	1.377(9)
C(23')-H(23')	0.9500	C(60)-C(61)	1.413(8)
C(24')-C(25')	1.362(18)	C(60)-H(60)	0.9500
C(24')-H(24')	0.9500	C(61)-C(62)	1.372(9)
C(25')-H(25')	0.9500	C(61)-O(6)	1.383(7)
C(8)-C(26)	1.379(8)	C(62)-H(62)	0.9500
C(8)-C(30)	1.400(8)	O(5)-C(63)	1.410(8)
C(26)-C(27)	1.407(9)	C(63)-H(63A)	0.9800
C(26)-H(26)	0.9500	C(63)-H(63B)	0.9800
C(27)-C(28)	1.361(10)	C(63)-H(63C)	0.9800
C(27)-H(27)	0.9500	O(6)-C(64)	1.457(7)
C(28)-C(29)	1.386(9)	C(64)-H(64A)	0.9800
C(28)-C(31)	1.518(10)	C(64)-H(64B)	0.9800
C(29)-C(30)	1.387(9)	C(64)-H(64C)	0.9800
C(29)-H(29)	0.9500	C(65)-O(7)	1.379(7)
C(30)-H(30)	0.9500	C(65)-C(66)	1.386(7)
C(31)-H(31A)	0.9800	C(66)-C(67)	1.372(9)
C(31)-H(31B)	0.9800	C(66)-H(66A)	0.9500
C(31)-H(31C)	0.9800	C(67)-O(8')	1.392(11)
N(3)-C(54)	1.400(8)	C(67)-O(8)	1.394(7)
N(3)-C(51)	1.486(7)	C(67)-C(68)	1.398(9)
N(3)-H(3)	0.97(5)	C(68)-H(68A)	0.9500
N(4)-C(55)	1.378(7)	O(7)-C(69)	1.444(7)
N(4)-C(52)	1.477(7)	C(69)-H(69A)	0.9800
N(4)-H(4)	0.97(6)	C(69)-H(69B)	0.9800
C(51)-C(57)	1.504(10)	C(69)-H(69C)	0.9800
C(51)-C(56)	1.510(8)	O(8)-C(70)	1.425(10)
C(51)-C(52)	1.619(8)	C(70)-H(70A)	0.9800
C(52)-C(53)	1.486(8)	C(70)-H(70B)	0.9800
C(52)-C(58)	1.519(8)	C(70)-H(70C)	0.9800
C(53)-C(54)	1.388(8)	O(8')-C(70')	1.422(13)
C(53)-C(59)	1.391(9)	C(70')-H(70D)	0.9800
C(54)-C(62)	1.385(9)	C(70')-H(70E)	0.9800
C(55)-C(68)	1.378(8)	C(70')-H(70F)	0.9800
C(55)-C(56)	1.400(7)	C(57)-C(75)	1.380(14)
C(56)-C(65)	1.390(8)	C(57)-C(71)	1.406(13)
C(59)-O(5)	1.363(6)	C(71)-C(72)	1.403(15)

C(71)-H(71)	0.9500	C(1)-N(1)-H(1)	113(4)
C(72)-C(73)	1.354(15)	C(5)-N(2)-C(2)	107.2(5)
C(72)-H(72)	0.9500	C(5)-N(2)-H(2)	106(3)
C(73)-C(74)	1.378(14)	C(2)-N(2)-H(2)	115(3)
C(73)-C(81)	1.541(16)	N(1)-C(1)-C(6)	113.2(5)
C(74)-C(75)	1.416(14)	N(1)-C(1)-C(7)	109.5(7)
C(74)-H(74)	0.9500	C(6)-C(1)-C(7)	114.2(8)
C(75)-H(75)	0.9500	N(1)-C(1)-C(7')	119.1(17)
C(71')-C(72')	1.403(15)	C(6)-C(1)-C(7')	104.8(19)
C(71')-H(71')	0.9500	N(1)-C(1)-C(2)	103.1(5)
C(72')-C(73')	1.354(16)	C(6)-C(1)-C(2)	101.3(4)
C(72')-H(72')	0.9500	C(7)-C(1)-C(2)	115.0(11)
C(73')-C(74')	1.391(15)	C(7')-C(1)-C(2)	114(3)
C(73')-H(73')	0.9500	N(2)-C(2)-C(8)	110.9(5)
C(74')-C(75')	1.416(14)	N(2)-C(2)-C(3)	110.7(5)
C(74')-H(74')	0.9500	C(8)-C(2)-C(3)	114.1(5)
C(75')-H(75')	0.9500	N(2)-C(2)-C(1)	103.8(4)
C(58)-C(80)	1.378(8)	C(8)-C(2)-C(1)	116.0(5)
C(58)-C(76)	1.393(8)	C(3)-C(2)-C(1)	100.5(5)
C(76)-C(77)	1.367(10)	C(4)-C(3)-C(9)	120.3(6)
C(76)-H(76A)	0.9500	C(4)-C(3)-C(2)	110.5(5)
C(77)-C(78)	1.384(11)	C(9)-C(3)-C(2)	129.2(5)
C(77)-H(77A)	0.9500	C(3)-C(4)-N(1)	111.4(5)
C(78)-C(79)	1.379(10)	C(3)-C(4)-C(12)	121.7(6)
C(78)-C(81')	1.530(14)	N(1)-C(4)-C(12)	126.9(6)
C(78)-H(78)	0.9500	C(6)-C(5)-C(18)	123.3(6)
C(79)-C(80)	1.397(9)	C(6)-C(5)-N(2)	111.7(5)
C(79)-H(79A)	0.9500	C(18)-C(5)-N(2)	125.0(6)
C(80)-H(80A)	0.9500	C(5)-C(6)-C(15)	119.2(5)
C(81)-H(81A)	0.9800	C(5)-C(6)-C(1)	111.8(5)
C(81)-H(81B)	0.9800	C(15)-C(6)-C(1)	128.9(5)
C(81)-H(81C)	0.9800	O(1)-C(9)-C(3)	114.3(5)
C(81')-H(81D)	0.9800	O(1)-C(9)-C(10)	126.1(6)
C(81')-H(81E)	0.9800	C(3)-C(9)-C(10)	119.6(6)
C(81')-H(81F)	0.9800	C(11)-C(10)-C(9)	119.4(6)
C(4)-N(1)-C(1)	109.0(5)	C(11)-C(10)-H(10)	120.3
C(4)-N(1)-H(1)	127(4)	C(9)-C(10)-H(10)	120.3

O(2)-C(11)-C(10)	113.6(6)	H(19A)-C(19)-H(19B)	109.5
O(2)-C(11)-C(12)	124.2(6)	O(3)-C(19)-H(19C)	109.5
C(10)-C(11)-C(12)	122.2(6)	H(19A)-C(19)-H(19C)	109.5
C(11)-C(12)-C(4)	116.8(6)	H(19B)-C(19)-H(19C)	109.5
C(11)-C(12)-H(12)	121.6	C(17)-O(4)-C(20)	115.1(8)
C(4)-C(12)-H(12)	121.6	C(17)-O(4')-C(20')	119.9(9)
C(9)-O(1)-C(13)	116.8(5)	C(21)-C(7)-C(25)	119.2(8)
O(1)-C(13)-H(13A)	109.5	C(21)-C(7)-C(1)	123.1(10)
O(1)-C(13)-H(13B)	109.5	C(25)-C(7)-C(1)	117.6(9)
H(13A)-C(13)-H(13B)	109.5	C(7)-C(21)-C(22)	119.4(9)
O(1)-C(13)-H(13C)	109.5	C(7)-C(21)-H(21)	120.3
H(13A)-C(13)-H(13C)	109.5	C(22)-C(21)-H(21)	120.3
H(13B)-C(13)-H(13C)	109.5	C(21)-C(22)-C(23)	121.3(8)
C(11)-O(2)-C(14)	118.6(6)	C(21)-C(22)-H(22)	119.4
O(2)-C(14)-H(14A)	109.5	C(23)-C(22)-H(22)	119.4
O(2)-C(14)-H(14B)	109.5	C(24)-C(23)-C(22)	117.6(8)
H(14A)-C(14)-H(14B)	109.5	C(24)-C(23)-H(23)	121.2
O(2)-C(14)-H(14C)	109.5	C(22)-C(23)-H(23)	121.2
H(14A)-C(14)-H(14C)	109.5	C(25)-C(24)-C(23)	121.9(9)
H(14B)-C(14)-H(14C)	109.5	C(25)-C(24)-H(24)	119.0
O(3)-C(15)-C(16)	124.4(5)	C(23)-C(24)-H(24)	119.0
O(3)-C(15)-C(6)	115.6(5)	C(24)-C(25)-C(7)	120.5(8)
C(16)-C(15)-C(6)	120.0(5)	C(24)-C(25)-H(25)	119.8
C(15)-C(16)-C(17)	118.1(5)	C(7)-C(25)-H(25)	119.8
C(15)-C(16)-H(16)	121.0	C(21')-C(7')-C(25')	120.1(18)
C(17)-C(16)-H(16)	121.0	C(21')-C(7')-C(1)	115(3)
C(18)-C(17)-C(16)	124.2(5)	C(25')-C(7')-C(1)	124(3)
C(18)-C(17)-O(4)	124.6(7)	C(7')-C(21')-C(22')	119(2)
C(16)-C(17)-O(4)	109.1(7)	C(7')-C(21')-H(21')	120.6
C(18)-C(17)-O(4')	120.1(7)	C(22')-C(21')-H(21')	120.6
C(16)-C(17)-O(4')	114.6(7)	C(23')-C(22')-C(21')	121(2)
C(17)-C(18)-C(5)	115.0(6)	C(23')-C(22')-H(22')	119.7
C(17)-C(18)-H(18)	122.5	C(21')-C(22')-H(22')	119.7
C(5)-C(18)-H(18)	122.5	C(24')-C(23')-C(22')	119(2)
C(15)-O(3)-C(19)	116.2(4)	C(24')-C(23')-H(23')	120.7
O(3)-C(19)-H(19A)	109.5	C(22')-C(23')-H(23')	120.7
O(3)-C(19)-H(19B)	109.5	C(23')-C(24')-C(25')	122(2)

C(23')-C(24')-H(24')	119.2	C(57)-C(51)-C(56)	111.1(5)
C(25')-C(24')-H(24')	119.2	N(3)-C(51)-C(52)	103.7(4)
C(24')-C(25')-C(7')	119.4(19)	C(57)-C(51)-C(52)	115.5(5)
C(24')-C(25')-H(25')	120.3	C(56)-C(51)-C(52)	99.9(4)
C(7')-C(25')-H(25')	120.3	N(4)-C(52)-C(53)	113.5(5)
C(26)-C(8)-C(30)	119.7(6)	N(4)-C(52)-C(58)	111.2(5)
C(26)-C(8)-C(2)	119.4(5)	C(53)-C(52)-C(58)	114.0(4)
C(30)-C(8)-C(2)	120.9(5)	N(4)-C(52)-C(51)	103.6(4)
C(8)-C(26)-C(27)	118.5(6)	C(53)-C(52)-C(51)	100.4(5)
C(8)-C(26)-H(26)	120.7	C(58)-C(52)-C(51)	113.3(5)
C(27)-C(26)-H(26)	120.7	C(54)-C(53)-C(59)	119.3(6)
C(28)-C(27)-C(26)	123.4(6)	C(54)-C(53)-C(52)	112.2(5)
C(28)-C(27)-H(27)	118.3	C(59)-C(53)-C(52)	128.4(5)
C(26)-C(27)-H(27)	118.3	C(62)-C(54)-C(53)	122.1(6)
C(27)-C(28)-C(29)	116.8(6)	C(62)-C(54)-N(3)	127.4(6)
C(27)-C(28)-C(31)	120.4(7)	C(53)-C(54)-N(3)	110.4(5)
C(29)-C(28)-C(31)	122.9(7)	C(68)-C(55)-N(4)	127.6(5)
C(28)-C(29)-C(30)	122.4(7)	C(68)-C(55)-C(56)	121.8(5)
C(28)-C(29)-H(29)	118.8	N(4)-C(55)-C(56)	110.3(5)
C(30)-C(29)-H(29)	118.8	C(65)-C(56)-C(55)	119.5(5)
C(29)-C(30)-C(8)	119.2(6)	C(65)-C(56)-C(51)	128.9(5)
C(29)-C(30)-H(30)	120.4	C(55)-C(56)-C(51)	111.4(5)
C(8)-C(30)-H(30)	120.4	O(5)-C(59)-C(60)	123.6(6)
C(28)-C(31)-H(31A)	109.5	O(5)-C(59)-C(53)	116.0(5)
C(28)-C(31)-H(31B)	109.5	C(60)-C(59)-C(53)	120.4(6)
H(31A)-C(31)-H(31B)	109.5	C(59)-C(60)-C(61)	118.5(6)
C(28)-C(31)-H(31C)	109.5	C(59)-C(60)-H(60)	120.8
H(31A)-C(31)-H(31C)	109.5	C(61)-C(60)-H(60)	120.8
H(31B)-C(31)-H(31C)	109.5	C(62)-C(61)-O(6)	124.2(5)
C(54)-N(3)-C(51)	107.9(5)	C(62)-C(61)-C(60)	122.4(6)
C(54)-N(3)-H(3)	110(3)	O(6)-C(61)-C(60)	113.4(6)
C(51)-N(3)-H(3)	115(3)	C(61)-C(62)-C(54)	117.4(6)
C(55)-N(4)-C(52)	109.6(5)	C(61)-C(62)-H(62)	121.3
C(55)-N(4)-H(4)	120(3)	C(54)-C(62)-H(62)	121.3
C(52)-N(4)-H(4)	111(3)	C(59)-O(5)-C(63)	117.9(5)
N(3)-C(51)-C(57)	112.4(5)	O(5)-C(63)-H(63A)	109.5
N(3)-C(51)-C(56)	113.5(5)	O(5)-C(63)-H(63B)	109.5

H(63A)-C(63)-H(63B)	109.5	C(72)-C(71)-C(57)	122.9(13)
O(5)-C(63)-H(63C)	109.5	C(72)-C(71)-H(71)	118.5
H(63A)-C(63)-H(63C)	109.5	C(57)-C(71)-H(71)	118.5
H(63B)-C(63)-H(63C)	109.5	C(73)-C(72)-C(71)	120.5(13)
C(61)-O(6)-C(64)	115.0(5)	C(73)-C(72)-H(72)	119.7
O(6)-C(64)-H(64A)	109.5	C(71)-C(72)-H(72)	119.7
O(6)-C(64)-H(64B)	109.5	C(72)-C(73)-C(74)	119.8(13)
H(64A)-C(64)-H(64B)	109.5	C(72)-C(73)-C(81)	121.1(11)
O(6)-C(64)-H(64C)	109.5	C(74)-C(73)-C(81)	119.1(12)
H(64A)-C(64)-H(64C)	109.5	C(73)-C(74)-C(75)	118.1(14)
H(64B)-C(64)-H(64C)	109.5	C(73)-C(74)-H(74)	121.0
O(7)-C(65)-C(66)	125.0(5)	C(75)-C(74)-H(74)	121.0
O(7)-C(65)-C(56)	115.7(5)	C(57)-C(75)-C(74)	124.8(13)
C(66)-C(65)-C(56)	119.3(5)	C(57)-C(75)-H(75)	117.6
C(67)-C(66)-C(65)	120.1(5)	C(74)-C(75)-H(75)	117.6
C(67)-C(66)-H(66A)	119.9	C(72')-C(71')-H(71')	119.9
C(65)-C(66)-H(66A)	119.9	C(73')-C(72')-C(71')	119.3(13)
C(66)-C(67)-O(8')	113.1(13)	C(73')-C(72')-H(72')	120.4
C(66)-C(67)-O(8)	115.1(6)	C(71')-C(72')-H(72')	120.4
C(66)-C(67)-C(68)	122.0(5)	C(72')-C(73')-C(74')	121.2(12)
O(8')-C(67)-C(68)	123.8(13)	C(72')-C(73')-H(73')	119.4
O(8)-C(67)-C(68)	122.2(7)	C(74')-C(73')-H(73')	119.4
C(55)-C(68)-C(67)	117.2(6)	C(73')-C(74')-C(75')	119.4(14)
C(55)-C(68)-H(68A)	121.4	C(73')-C(74')-H(74')	120.3
C(67)-C(68)-H(68A)	121.4	C(75')-C(74')-H(74')	120.3
C(65)-O(7)-C(69)	117.0(4)	C(74')-C(75')-H(75')	120.2
O(7)-C(69)-H(69A)	109.5	C(80)-C(58)-C(76)	115.8(6)
O(7)-C(69)-H(69B)	109.5	C(80)-C(58)-C(52)	122.8(5)
H(69A)-C(69)-H(69B)	109.5	C(76)-C(58)-C(52)	121.3(6)
O(7)-C(69)-H(69C)	109.5	C(77)-C(76)-C(58)	123.9(7)
H(69A)-C(69)-H(69C)	109.5	C(77)-C(76)-H(76A)	118.1
H(69B)-C(69)-H(69C)	109.5	C(58)-C(76)-H(76A)	118.1
C(67)-O(8)-C(70)	117.4(8)	C(76)-C(77)-C(78)	119.0(6)
C(67)-O(8')-C(70')	115(2)	C(76)-C(77)-H(77A)	120.5
C(75)-C(57)-C(71)	113.6(10)	C(78)-C(77)-H(77A)	120.5
C(75)-C(57)-C(51)	115.8(8)	C(79)-C(78)-C(77)	119.4(7)
C(71)-C(57)-C(51)	130.6(8)	C(79)-C(78)-C(81')	125.4(10)

C(77)-C(78)-C(81')	115.2(9)	C(58)-C(80)-C(79)	121.9(6)
C(79)-C(78)-H(78)	120.3	C(58)-C(80)-H(80A)	119.1
C(77)-C(78)-H(78)	120.3	C(79)-C(80)-H(80A)	119.1
C(78)-C(79)-C(80)	120.0(7)	C(78)-C(81')-H(81D)	109.5
C(78)-C(79)-H(79A)	120.0	C(78)-C(81')-H(81E)	109.5
C(80)-C(79)-H(79A)	120.0	C(78)-C(81')-H(81F)	109.5

Table S65. Anisotropic displacement parameters ($Å^2$ x 10^3) for (+/-)-**8**. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [h^2 a^{*2} U_{11} + ... + 2 h k a* b* U_{12}]

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	31(2)	49(4)	19(3)	8(3)	-1(2)	-9(2)
N2	26(1)	29(1)	22(1)	-1(2)	1(1)	0(2)
C1	26(1)	34(1)	19(1)	-3(3)	2(1)	2(2)
C2	24(2)	35(3)	26(3)	6(3)	-4(2)	3(2)
C3	24(1)	32(1)	26(1)	-2(3)	3(1)	-2(2)
C4	22(1)	41(1)	25(1)	3(3)	6(1)	1(2)
C5	25(2)	26(3)	30(3)	-4(3)	3(2)	2(2)
C6	25(1)	25(1)	24(1)	-1(2)	1(1)	-1(2)
C9	27(2)	32(3)	23(3)	3(3)	2(2)	0(2)
C10	31(3)	27(3)	30(3)	-1(3)	4(2)	-5(2)
C11	27(3)	47(4)	28(3)	-4(3)	8(2)	-4(3)
C12	25(1)	47(1)	23(1)	-3(3)	3(1)	2(2)
O1	37(1)	31(1)	23(1)	0(2)	0(1)	-1(2)
C13	56(4)	36(4)	35(4)	9(3)	8(3)	-3(3)
O2	43(2)	46(3)	30(2)	-4(2)	0(2)	-10(2)
C14	89(3)	54(3)	33(1)	0(3)	-14(2)	13(3)
C15	31(1)	23(1)	23(1)	1(2)	0(1)	0(2)
C16	35(3)	35(3)	22(3)	6(2)	8(2)	5(2)
C17	39(3)	39(4)	53(4)	19(3)	21(3)	18(3)
C18	33(3)	33(3)	35(3)	13(3)	10(2)	14(2)
O3	37(1)	35(1)	23(1)	0(2)	3(1)	1(2)
C19	49(3)	42(4)	23(3)	9(3)	16(3)	1(3)
O4	31(3)	52(6)	27(4)	7(3)	8(3)	11(4)
C20	38(5)	50(6)	48(6)	7(4)	8(4)	24(4)
O4'	31(3)	52(6)	27(4)	7(3)	8(3)	11(4)
C20'	38(5)	50(6)	48(6)	7(4)	8(4)	24(4)
C7	19(6)	27(3)	20(4)	4(3)	6(6)	6(3)
C21	24(6)	49(5)	35(7)	17(4)	4(4)	16(4)
C22	26(5)	49(4)	45(4)	12(4)	4(5)	20(4)
C23	47(8)	28(4)	33(5)	6(3)	14(6)	15(5)
C24	28(6)	39(4)	37(5)	-6(4)	4(5)	5(5)
C25	26(6)	30(4)	25(4)	-2(3)	0(5)	7(4)
C7'	19(6)	27(3)	20(4)	4(3)	6(6)	6(3)

C21'	24(6)	49(5)	35(7)	17(4)	4(4)	16(4)
C22'	26(5)	49(4)	45(4)	12(4)	4(5)	20(4)
C23'	47(8)	28(4)	33(5)	6(3)	14(6)	15(5)
C24'	28(6)	39(4)	37(5)	-6(4)	4(5)	5(5)
C25'	26(6)	30(4)	25(4)	-2(3)	0(5)	7(4)
C8	35(1)	31(1)	24(1)	-1(3)	6(1)	0(2)
C26	39(1)	46(1)	38(1)	-7(3)	11(1)	-1(3)
C27	50(4)	41(4)	54(4)	2(4)	26(3)	11(3)
C28	67(4)	28(3)	42(4)	-1(3)	32(3)	-3(3)
C29	74(2)	33(1)	32(1)	1(3)	13(1)	-1(4)
C30	45(1)	32(1)	31(1)	2(3)	9(1)	3(3)
C31	108(7)	65(6)	89(7)	-16(5)	63(6)	-11(5)
N3	27(2)	34(3)	28(3)	-4(2)	2(2)	-2(2)
N4	26(1)	29(1)	22(1)	-1(2)	1(1)	0(2)
C51	26(1)	34(1)	19(1)	-3(3)	2(1)	2(2)
C52	28(2)	24(3)	19(3)	-1(2)	5(2)	-2(2)
C53	24(1)	32(1)	26(1)	-2(3)	3(1)	-2(2)
C54	22(1)	41(1)	25(1)	3(3)	6(1)	1(2)
C55	30(2)	22(3)	19(3)	-2(2)	5(2)	5(2)
C56	25(1)	25(1)	24(1)	-1(2)	1(1)	-1(2)
C59	26(2)	36(4)	22(3)	0(3)	1(2)	2(2)
C60	31(3)	48(4)	24(3)	-1(3)	4(2)	8(3)
C61	26(2)	39(4)	30(3)	7(3)	3(2)	7(3)
C62	25(1)	47(1)	23(1)	-3(3)	3(1)	2(2)
O5	37(1)	31(1)	23(1)	0(2)	0(1)	-1(2)
C63	65(4)	38(4)	26(3)	-7(3)	-8(3)	18(3)
O6	46(2)	54(3)	25(2)	5(2)	2(2)	23(2)
C64	89(3)	54(3)	33(1)	0(3)	-14(2)	13(3)
C65	31(1)	23(1)	23(1)	1(2)	0(1)	0(2)
C66	33(3)	25(3)	32(3)	-1(2)	9(2)	-1(2)
C67	30(3)	34(3)	40(4)	-3(3)	12(2)	-4(2)
C68	36(3)	31(3)	31(3)	-4(3)	3(2)	2(2)
O7	37(1)	35(1)	23(1)	0(2)	3(1)	1(2)
C69	56(4)	44(4)	29(4)	-5(3)	5(3)	-3(3)
O8	34(3)	31(5)	55(4)	-11(4)	16(3)	-10(3)
C70	47(6)	77(10)	79(8)	-49(7)	30(5)	-29(6)
O8'	34(3)	31(5)	55(4)	-11(4)	16(3)	-10(3)

C70'	47(6)	77(10)	79(8)	-49(7)	30(5)	-29(6)
C57	38(3)	36(4)	24(3)	-5(3)	12(3)	-8(3)
C71	52(6)	20(7)	33(5)	2(10)	9(6)	0(6)
C72	48(4)	24(3)	51(4)	-1(3)	15(3)	-4(3)
C73	40(8)	32(4)	28(6)	-6(4)	3(5)	-12(6)
C74	59(9)	25(5)	21(8)	-14(4)	5(5)	-8(5)
C75	48(4)	24(3)	51(4)	-1(3)	15(3)	-4(3)
C57'	38(3)	36(4)	24(3)	-5(3)	12(3)	-8(3)
C71'	52(6)	20(7)	33(5)	2(10)	9(6)	0(6)
C72'	48(4)	24(3)	51(4)	-1(3)	15(3)	-4(3)
C73'	40(8)	32(4)	28(6)	-6(4)	3(5)	-12(6)
C74'	59(9)	25(5)	21(8)	-14(4)	5(5)	-8(5)
C75'	48(4)	24(3)	51(4)	-1(3)	15(3)	-4(3)
C58	35(1)	31(1)	24(1)	-1(3)	6(1)	0(2)
C76	39(1)	46(1)	38(1)	-7(3)	11(1)	-1(3)
C77	46(3)	66(5)	36(4)	-13(4)	20(3)	-14(4)
C78	81(5)	38(4)	39(4)	-15(3)	27(4)	-23(4)
C79	74(2)	33(1)	32(1)	1(3)	13(1)	-1(4)
C80	45(1)	32(1)	31(1)	2(3)	9(1)	3(3)
C81	46(5)	20(4)	56(6)	4(4)	-8(4)	-8(4)
C81'	75(9)	24(6)	47(7)	-4(6)	43(7)	-1(6)

Table S66. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for (+/-)-**8**.

	x	y	z	$U_{(eq)}$
H1	4940(50)	-430(30)	730(50)	49
H2	7160(40)	-1320(30)	3960(40)	39
H10	4047	-2826	3427	35
H12	3665	-1657	521	38
H13A	5646	-2490	6054	63
H13B	5452	-2942	4909	63
H13C	4384	-2557	5413	63
H14A	2489	-3369	102	90
H14B	3604	-2908	-88	90
H14C	2385	-2535	13	90
H16	8985	-642	-86	36
H18	9145	-1463	3134	40
H19A	7027	483	-1479	56
H19B	7658	-262	-1447	56
H19C	8258	390	-782	56
H20A	11310	-2178	1568	68
H20B	10079	-2354	2012	68
H20C	10859	-1745	2608	68
H20D	11885	-1575	2114	68
H20E	10801	-1994	2522	68
H20F	11079	-1220	2989	68
H21	4196	648	1520	43
H22	4152	1863	1704	48
H23	5851	2489	2252	43
H24	7492	1859	2892	42
H25	7513	662	2848	33
H21'	4393	691	1818	43
H22'	4564	1911	1665	48
H23'	6243	2467	2446	43
H24'	7820	1794	3063	42
H25'	7782	594	2866	33
H26	3712	-483	3855	49
H27	2988	209	5278	57

H29	6163	827	6363	55
H30	6943	127	5005	43
H31A	4577	1226	7386	126
H31B	3453	741	7290	126
H31C	3515	1414	6487	126
H3	120(40)	3150(30)	600(50)	44
H4	2220(40)	3840(30)	4190(40)	39
H60	-825	5411	3416	41
H62	-1255	4232	517	38
H63A	694	5086	6019	65
H63B	-516	5189	5289	65
H63C	625	5542	4874	65
H64A	-2611	5810	-7	90
H64B	-2811	4995	215	90
H64C	-1619	5257	-223	90
H66A	4139	3151	5	35
H68A	4266	3968	3201	39
H69A	2090	2090	-1507	64
H69B	3320	2128	-795	64
H69C	2791	2811	-1428	64
H70A	6914	4206	2107	100
H70B	6220	3752	2966	100
H70C	5770	4515	2585	100
H70D	6407	4720	1704	100
H70E	6040	4237	2725	100
H70F	5172	4821	2187	100
H71	-597	1745	1553	42
H72	-393	546	1866	48
H74	2837	840	3253	42
H75	2627	2045	2910	48
H71'	-702	2014	1604	42
H72'	-843	784	1546	48
H73'	699	116	2254	40
H74'	2458	639	2862	42
H75'	2581	1877	2979	48
H76A	-1218	3036	3826	49
H77A	-2028	2348	5148	58

H78	-832	1679	6432	62
H79A	1164	1755	6388	55
H80A	1957	2442	5009	43
H81A	819	-459	2137	62
H81B	1428	-364	3397	62
H81C	2183	-341	2321	62
H81D	-1712	1824	7072	70
H81E	-578	1359	7338	70
H81F	-1518	1137	6333	70

Table S67. Torsion angles [°] for (+/-)-**8**.

C4-N1-C1-C6	84.9(6)	C18-C5-C6-C1	179.0(6)
C4-N1-C1-C7	-146.5(11)	N2-C5-C6-C1	-3.7(7)
C4-N1-C1-C7'	-151(3)	N1-C1-C6-C5	-119.0(6)
C4-N1-C1-C2	-23.6(6)	C7-C1-C6-C5	114.9(11)
C5-N2-C2-C8	-146.0(5)	C7'-C1-C6-C5	110(3)
C5-N2-C2-C3	86.2(6)	C2-C1-C6-C5	-9.3(6)
C5-N2-C2-C1	-20.8(6)	N1-C1-C6-C15	65.1(8)
N1-C1-C2-N2	135.2(5)	C7-C1-C6-C15	-61.0(12)
C6-C1-C2-N2	17.9(6)	C7'-C1-C6-C15	-66(3)
C7-C1-C2-N2	-105.8(7)	C2-C1-C6-C15	174.8(6)
C7'-C1-C2-N2	-94.1(16)	C4-C3-C9-O1	-179.1(5)
N1-C1-C2-C8	-102.9(5)	C2-C3-C9-O1	0.5(9)
C6-C1-C2-C8	139.8(5)	C4-C3-C9-C10	1.6(9)
C7-C1-C2-C8	16.2(8)	C2-C3-C9-C10	-178.8(5)
C7'-C1-C2-C8	27.8(16)	O1-C9-C10-C11	-178.7(5)
N1-C1-C2-C3	20.6(5)	C3-C9-C10-C11	0.5(9)
C6-C1-C2-C3	-96.7(5)	C9-C10-C11-O2	179.3(5)
C7-C1-C2-C3	139.7(6)	C9-C10-C11-C12	-1.6(9)
C7'-C1-C2-C3	151.3(16)	O2-C11-C12-C4	179.6(5)
N2-C2-C3-C4	-120.9(5)	C10-C11-C12-C4	0.5(8)
C8-C2-C3-C4	113.1(6)	C3-C4-C12-C11	1.6(8)
C1-C2-C3-C4	-11.7(6)	N1-C4-C12-C11	-178.3(5)
N2-C2-C3-C9	59.4(8)	C3-C9-O1-C13	179.8(5)
C8-C2-C3-C9	-66.6(8)	C10-C9-O1-C13	-1.0(8)
C1-C2-C3-C9	168.6(6)	C10-C11-O2-C14	-163.8(5)
C9-C3-C4-N1	177.2(5)	C12-C11-O2-C14	17.0(9)
C2-C3-C4-N1	-2.4(7)	C5-C6-C15-O3	-177.5(5)
C9-C3-C4-C12	-2.8(9)	C1-C6-C15-O3	-1.8(9)
C2-C3-C4-C12	177.6(5)	C5-C6-C15-C16	4.9(9)
C1-N1-C4-C3	17.5(7)	C1-C6-C15-C16	-179.5(6)
C1-N1-C4-C12	-162.6(5)	O3-C15-C16-C17	179.6(6)
C2-N2-C5-C6	16.2(7)	C6-C15-C16-C17	-3.0(9)
C2-N2-C5-C18	-166.5(6)	C15-C16-C17-C18	0.9(11)
C18-C5-C6-C15	-4.7(9)	C15-C16-C17-O4	165.2(7)
N2-C5-C6-C15	172.7(5)	C15-C16-C17-O4'	-167.4(8)

C16-C17-C18-C5	-0.5(11)	C21'-C7'-C25'-C24'	-3(11)
O4-C17-C18-C5	-162.4(8)	C1-C7'-C25'-C24'	-178(5)
O4'-C17-C18-C5	167.1(8)	N2-C2-C8-C26	-155.0(6)
C6-C5-C18-C17	2.5(10)	C3-C2-C8-C26	-29.2(8)
N2-C5-C18-C17	-174.6(6)	C1-C2-C8-C26	86.9(7)
C16-C15-O3-C19	-8.5(9)	N2-C2-C8-C30	27.0(8)
C6-C15-O3-C19	174.0(5)	C3-C2-C8-C30	152.9(6)
C18-C17-O4-C20	-6.9(14)	C1-C2-C8-C30	-91.0(7)
C16-C17-O4-C20	-171.1(9)	C30-C8-C26-C27	0.3(10)
C18-C17-O4'-C20'	0.8(15)	C2-C8-C26-C27	-177.6(6)
C16-C17-O4'-C20'	169.5(10)	C8-C26-C27-C28	0.4(11)
N1-C1-C7-C21	12(3)	C26-C27-C28-C29	-0.2(11)
C6-C1-C7-C21	140(2)	C26-C27-C28-C31	179.5(7)
C2-C1-C7-C21	-103(2)	C27-C28-C29-C30	-0.8(10)
N1-C1-C7-C25	-165.2(17)	C31-C28-C29-C30	179.5(7)
C6-C1-C7-C25	-37(2)	C28-C29-C30-C8	1.5(10)
C2-C1-C7-C25	79(2)	C26-C8-C30-C29	-1.3(10)
C25-C7-C21-C22	-3(4)	C2-C8-C30-C29	176.7(6)
C1-C7-C21-C22	-179.7(18)	C54-N3-C51-C57	149.1(5)
C7-C21-C22-C23	5(3)	C54-N3-C51-C56	-83.7(6)
C21-C22-C23-C24	-5(3)	C54-N3-C51-C52	23.7(6)
C22-C23-C24-C25	2(3)	C55-N4-C52-C53	-84.8(6)
C23-C24-C25-C7	0(3)	C55-N4-C52-C58	145.2(5)
C21-C7-C25-C24	0(4)	C55-N4-C52-C51	23.1(6)
C1-C7-C25-C24	177.3(18)	N3-C51-C52-N4	-136.6(5)
N1-C1-C7'-C21'	21(7)	C57-C51-C52-N4	100.0(6)
C6-C1-C7'-C21'	148(6)	C56-C51-C52-N4	-19.2(6)
C2-C1-C7'-C21'	-102(6)	N3-C51-C52-C53	-19.2(5)
N1-C1-C7'-C25'	-164(5)	C57-C51-C52-C53	-142.6(5)
C6-C1-C7'-C25'	-36(7)	C56-C51-C52-C53	98.2(5)
C2-C1-C7'-C25'	73(7)	N3-C51-C52-C58	102.8(5)
C25'-C7'-C21'-C22'	10(11)	C57-C51-C52-C58	-20.7(7)
C1-C7'-C21'-C22'	-175(5)	C56-C51-C52-C58	-139.9(5)
C7'-C21'-C22'-C23'	-11(10)	N4-C52-C53-C54	118.8(5)
C21'-C22'-C23'-C24'	5(9)	C58-C52-C53-C54	-112.5(6)
C22'-C23'-C24'-C25'	2(9)	C51-C52-C53-C54	8.9(6)
C23'-C24'-C25'-C7'	-3(10)	N4-C52-C53-C59	-58.4(8)

C58-C52-C53-C59	70.3(8)	C51-C56-C65-O7	0.7(9)
C51-C52-C53-C59	-168.3(6)	C55-C56-C65-C66	-1.7(9)
C59-C53-C54-C62	-0.2(8)	C51-C56-C65-C66	-177.1(6)
C52-C53-C54-C62	-177.7(5)	O7-C65-C66-C67	-178.3(6)
C59-C53-C54-N3	-176.8(5)	C56-C65-C66-C67	-0.7(9)
C52-C53-C54-N3	5.7(6)	C65-C66-C67-O8'	-165.1(14)
C51-N3-C54-C62	164.3(5)	C65-C66-C67-O8	173.8(7)
C51-N3-C54-C53	-19.3(6)	C65-C66-C67-C68	3.2(10)
C52-N4-C55-C68	168.5(6)	N4-C55-C68-C67	174.0(6)
C52-N4-C55-C56	-17.6(7)	C56-C55-C68-C67	0.7(9)
C68-C55-C56-C65	1.7(9)	C66-C67-C68-C55	-3.1(10)
N4-C55-C56-C65	-172.7(5)	O8'-C67-C68-C55	163.8(16)
C68-C55-C56-C51	177.9(6)	O8-C67-C68-C55	-173.1(8)
N4-C55-C56-C51	3.5(7)	C66-C65-O7-C69	3.0(8)
N3-C51-C56-C65	-64.2(8)	C56-C65-O7-C69	-174.7(5)
C57-C51-C56-C65	63.6(8)	C66-C67-O8-C70	-170.6(9)
C52-C51-C56-C65	-174.0(6)	C68-C67-O8-C70	0.1(12)
N3-C51-C56-C55	120.0(5)	C66-C67-O8'-C70'	169(2)
C57-C51-C56-C55	-112.1(6)	C68-C67-O8'-C70'	1(3)
C52-C51-C56-C55	10.2(6)	N3-C51-C57-C75	167(2)
C54-C53-C59-O5	-177.5(5)	C56-C51-C57-C75	39(2)
C52-C53-C59-O5	-0.5(9)	C52-C51-C57-C75	-74(2)
C54-C53-C59-C60	0.2(9)	N3-C51-C57-C71	-12(3)
C52-C53-C59-C60	177.2(5)	C56-C51-C57-C71	-140(3)
O5-C59-C60-C61	177.0(5)	C52-C51-C57-C71	107(3)
C53-C59-C60-C61	-0.5(9)	C75-C57-C71-C72	2(6)
C59-C60-C61-C62	0.8(9)	C51-C57-C71-C72	-179(2)
C59-C60-C61-O6	-178.6(5)	C57-C71-C72-C73	-3(6)
O6-C61-C62-C54	178.5(5)	C71-C72-C73-C74	5(4)
C60-C61-C62-C54	-0.9(9)	C71-C72-C73-C81	-175(3)
C53-C54-C62-C61	0.6(8)	C72-C73-C74-C75	-5(3)
N3-C54-C62-C61	176.5(5)	C81-C73-C74-C75	175(2)
C60-C59-O5-C63	3.4(8)	C71-C57-C75-C74	-2(5)
C53-C59-O5-C63	-179.0(6)	C51-C57-C75-C74	178(3)
C62-C61-O6-C64	0.2(8)	C73-C74-C75-C57	4(5)
C60-C61-O6-C64	179.6(5)	C71'-C72'-C73'-C74'	-2(4)
C55-C56-C65-O7	176.2(5)	C72'-C73'-C74'-C75'	4(3)

N4-C52-C58-C80	-24.4(8)	C58-C76-C77-C78	-0.2(11)
C53-C52-C58-C80	-154.2(6)	C76-C77-C78-C79	1.0(11)
C51-C52-C58-C80	91.8(7)	C76-C77-C78-C81'	179.8(8)
N4-C52-C58-C76	158.7(6)	C77-C78-C79-C80	-1.7(10)
C53-C52-C58-C76	28.9(8)	C81'-C78-C79-C80	179.6(8)
C51-C52-C58-C76	-85.1(7)	C76-C58-C80-C79	-0.6(9)
C80-C58-C76-C77	-0.1(10)	C52-C58-C80-C79	-177.6(6)
C52-C58-C76-C77	177.0(6)	C78-C79-C80-C58	1.5(10)

Table S68. Hydrogen bonds and close contacts for (+/-)-**8** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1...O3	0.84(6)	2.70(6)	3.177(6)	117(4)
N1-H1...O8#1	0.84(6)	2.91(6)	3.542(12)	133(5)
N1-H1...O8'#1	0.84(6)	2.54(6)	3.22(3)	138(5)
N2-H2...O1	0.90(6)	2.52(6)	3.056(6)	119(4)
N3-H3...O7	0.97(5)	2.65(5)	3.190(6)	115(4)
N3-H3...O4#2	0.97(5)	2.12(5)	3.043(10)	158(5)
N3-H3...O4'#2	0.97(5)	2.63(5)	3.502(11)	149(4)
N4-H4...O5	0.97(6)	2.45(5)	3.078(6)	122(4)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y-1/2,-z #2 -x+1,y+1/2,-z