Electronic Supplementary Information

Induction of chirality in 4,4'-azopyridine by halogen-bonding interaction with optically active ditopic donors

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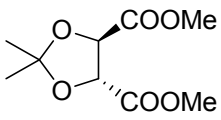
Experimental procedures

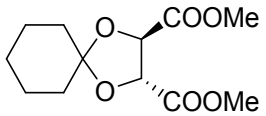
(4*R*,5*R*)-(–)-2,2-Dimethyl-1,3-dioxolane-4,5-dicarboxylic acid dimethyl ester ((*R*,*R*)-6a)



Dimethyl L-tartrate (10.4 g, 58.4 mmol) and *p*-toluenesulfonic acid monohydrate (200 mg, 1.1 mmol) were dissolved in benzene (200 mL) and then 2,2-dimethoxypropane (9.66 g, 92.8 mmol, 11.4 mL) was added. The mixture was refluxed for 18 h using Dean-Stark trap. After completion of the reaction, the mixture was cooled to room temperature and washed with aqueous solution of $NaHCO_3$ (2x25 mL) and next with water (2x25 mL). Aqueous layer was extracted with AcOEt (2x20 mL). All organic layers were combined, washed with water (2x25 mL) and brine (2x25 mL) and dried over anhydrous $MgSO_4$. Solvents were evaporated under reduced pressure to give acetal **(*R*,*R*)-6a** (11.65 g, 92%) as pale yellow oil which was used in the next step without further purification.[1] The ^{1}H NMR spectral data are consistent with those already reported in the literature.[2]

$[\alpha]^{22}_D$ –53 (c 1.0 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.83 (s, 2H), 3.85 (s, 6H), 1.52 (s, 6H).

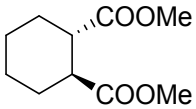
(2*R*,3*R*)-(–)-1,4-Dioxaspiro[4.5]decane-2,3-dicarboxylic acid dimethyl ester ((*R*,*R*)-6b)



Dimethyl L-tartrate (10.05 g, 56.4 mmol), cyclohexanone (27.68 g, 282 mmol, 29.1 mL) and *p*-toluenesulfonic acid monohydrate (100 mg, 0.5 mmol) were dissolved in toluene (200 mL). The mixture was refluxed for 96 h using Dean-Stark trap. After completion of the reaction, the mixture was cooled to room temperature and washed with aqueous solution of $NaHCO_3$ (2x25 mL) and water (2x25 mL). Organic layer was dried over anhydrous $MgSO_4$ and concentrated in vacuo. Crude product was purified by column chromatography on silica gel using hexane/AcOEt mixture (8:2 v/v) as the eluent (TLC R_f = 0.4) to give acetal **(*R*,*R*)-6b** (5.25 g, 36%) as pale yellow oil. The ^{1}H NMR spectral data are consistent with those already reported in the literature.[3]

$[\alpha]^{22}_D$ –26 (c 1.0 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.84 (s, 2H), 3.84 (s, 6H), 1.77-1.61 (m, 8H), 1.48-1.39 (m, 2H).

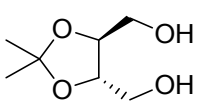
(1*S*,2*S*)-(+)-Cyclohexane-1,2-dicarboxylic acid dimethyl ester ((*S*,*S*)-7)



Diester **(*S*,*S*)-7** was synthesized following the literature method.[4]

$[\alpha]^{22}_D$ +20.5 (c 1.08 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.68 (s, 6H), 2.69-2.57 (m, 2H), 2.13-2.02 (m, 2H), 1.86-1.74 (m, 2H), 1.44-1.22 (m, 4H).

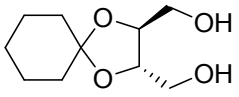
(4*S*,5*S*)-(–)-2,2-Dimethyl-4,5-di(hydroxymethyl)-1,3-dioxolane ((*S*,*S*)-4a)



To a suspension of $LiAlH_4$ (260 mg, 6.9 mmol) in dry THF (10 mL) cooled with ice bath a solution of acetal **(*R*,*R*)-6a** (1.00 g, 4.6 mmol) in dry THF (5 mL) was added dropwise over a period of 5 min. The ice bath was removed and the mixture was stirred at room temperature for 48 h. Then, the access of reducing agent was decomposed with THF/H_2O (10:1 v/v). The white precipitate was filtered out and washed with small amount of AcOEt. To the filtrate water (50 mL) and conc. HCl was added to pH around 4-5. Organic layer was separated, washed with brine (10 mL) and dried over anhydrous $MgSO_4$. Resulting solution was concentrated in vacuo to give diol **(*S*,*S*)-4a** (0.657 g, 88%) as pale yellow oil. The ^{1}H NMR spectral data are consistent with those already reported in the literature.[2]

$[\alpha]^{22}_D$ –2 (c 1.0 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.05-4.00 (m, 2H), 3.85-3.79 (ddd, *J* = 11.8; 2.6; 1.4 Hz, 2H), 3.79-3.69 (ddd, *J* = 11.8; 2.6; 1.4 Hz, 2H), 2.39 (bs, 2H), 1.45 (s, 6H).

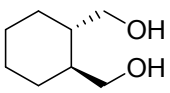
(2*S*,3*S*)-(–)-2,3-Di(hydroxymethyl)-1,4-dioxaspiro[4.5]decane ((*S*,*S*)-4b)



To a suspension of $LiAlH_4$ (420 mg, 11.1 mmol) in dry THF (15 mL) cooled with ice bath a solution of acetal **(*R*,*R*)-6b** (1.55 g, 6.0 mmol) in dry THF (15 mL) was added dropwise over a period of 5 min. The ice bath was removed and the mixture was stirred at room temperature for 72 h. Then, the access of reducing agent was decomposed with THF/H_2O (4:1 v/v). The white precipitate was filtered out and washed with THF (2x10 mL) and CH_2Cl_2 (2x10 mL). The filtrate was concentrated in vacuo and then brine (20 mL) was added. Resulting mixture was extracted with CH_2Cl_2 (4x20 mL). The organic layers were combined and dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure to give diol **(*S*,*S*)-4b** (1.158 g, 95%) as pale yellow oil.

$[\alpha]^{22}_D$ –8 (c 1.0 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.06-3.99 (m, 2H), 3.87-3.77 (m, 2H), 3.77-3.66 (d, 2H), 2.26 (s, 2H), 1.69-1.57 (m, 8H), 1.46-1.38 (m, 2H).

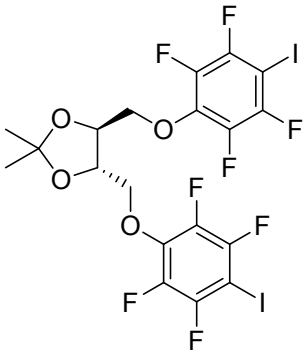
(1*S*,2*S*)-(–)-1,2-di(hydroxymethyl)cyclohexane ((*S*,*S*)-5)



To a suspension of $LiAlH_4$ (2,75 g, 72.46 mmol) in dry THF (20 mL) cooled with ice bath a solution of diester **(*S*,*S*)-7** (3.59 g, 17.93 mmol) in dry THF (50 mL) was added dropwise over a period of 15 min. The ice bath was removed and the mixture was stirred at room temperature for 3 h. Then, the access of reducing agent was decomposed with THF/H_2O (4:1 v/v) and 4M NaOH (5 mL). The white precipitate was filtered out and washed with THF (3x10 mL) and the filtrate was concentrated *in vacuo*. To the residue brine was added (25 mL) and the mixture was extracted with Et_2O (4x20 mL). The organic layers were combined and dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure to give diol **(*S*,*S*)-5** (2.06 g, 80%) as colourless oil which solidified upon storage (mp 53-55°C). The properties and spectral data are consistent with those already reported in the literature.[5]

$[\alpha]^{22}_D$ –14 (c 0.5 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.84 (bs, 2H), 3.61 (dd, *J* = 11.2; 2.8 Hz, 2H), 3.51 (dd, *J* = 11.1; 6.8 Hz, 2H), 1.82-1.68 (m, 2H), 1.68-1.57 (m, 2H), 1.39-1.29 (m, 2H), 1.29-1.16 (m, 2H), 1.11-0.95 (m, 2H).

(4*S*,5*S*)-(–)-2,2-dimethyl-4,5-di(2,3,5,6-tetrafluoro-4-iodophenoxymethyl)-1,3-dioxolane ((*S*,*S*)-1a)



Compound **(*S*,*S*)-1a** was prepared following procedure found in the literature.[6] Diol **(*S*,*S*)-4a** (0.5 g, 3.08 mmol), pentafluoroiodobenzene (4.33 g, 1.96 mL, 14.73 mmol) and Cs_2CO_3 (2.51 g, 7.70 mmol) was stirred and heated at 150°C for 5 h. Then, the mixture was cooled to room temperature and water (10 mL) was added. It was then stirred until inorganic salts were dissolved. Organic layer was separated and aqueous layer was extracted with $CHCl_3$ (3x4 mL). All organic layers were combined, washed with water (5 mL) and dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure to give crude product which was purified by column chromatography on silica gel using hexane/AcOEt mixture (9:1 v/v) as the eluent to give product **(*S*,*S*)-1a** (1.19 g, 55%) as colourless oil which solidifies if kept in a refrigerator.

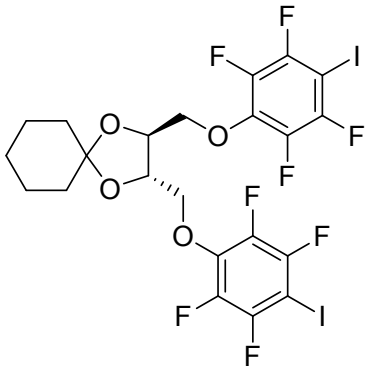
$[\alpha]^{22}_D$ –20 (c 0.6 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.57-4.43 (m, 4H), 4.43-4.37 (m, 2H), 1.47 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.64, 146.21, 141.75, 139.10, 137.84, 110.66, 76.23, 74.07, 64.46 (t, J_{CF} = 28.1 Hz) 26.74. ^{19}F NMR (376 MHz, $CDCl_3$): δ -120.87, -154.16.

Complex with apy (**apy·(*S*,*S*)-1a**): mp 100-102°C

General procedure for preparation of halogen bond donors (*S*,*S*)-1b, (*S*,*S*)-2 and (*R*)-3:

Procedure is analogous to that found in the literature.[7] To a suspension of NaH (5 eq) in dry DMF cooled with ice bath a solution of suitable diol (**(*S*,*S*)-4b**, **(*S*,*S*)-5** or (*R*)-BINOL) (1 eq) in dry DMF was added dropwise over a period of 15 min and the mixture was stirred at room temperature for 45 min. Then, solution of pentafluoroiodobenzene (4 eq) in dry DMF was added and the mixture was stirred for 24 h. Next, water was added to decompose excess of NaH and the mixture was extracted with CH_2Cl_2. The organic layers were combined, dried over anhydrous $MgSO_4$ and the solvent was removed *in vacuo*. The crude product was purified by column chromatography on silica gel using hexane/AcOEt mixture (15:1 v/v) as the eluent to give pure compounds **(*S*,*S*)-1b**, **(*S*,*S*)-2** and **(*R*)-3**.

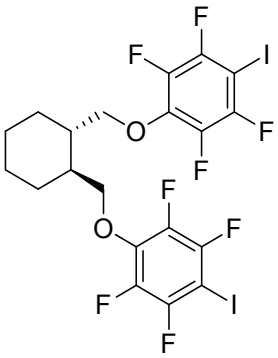
(2*S*,3*S*)-(–)-2,3-di(2,3,5,6-tetrafluoro-4-iodophenoxymethyl)-1,4-dioxaspiro[4.5]decane ((*S*,*S*)-1b)



Yield: 71%, colourless oil.
$[\alpha]^{22}_{D}$ –12 (c 0.5 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.55-4.42 (m, 4H), 4.42-4.34 (m, 2H), 1.72-1.61 (m, 4H), 1.61-1.50 (m, 4H), 1.44-1.34 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.51, 146.08, 141.77, 139.28, 137.93, 111.27, 75.99, 74.33, 64.38 (t, J_{CF} = 28.1 Hz), 36.29, 24.93, 23.73. ^{19}F NMR (376 MHz, $CDCl_3$): δ -121.02, -154.07.

Complex with apy (**apy·(*S*,*S*)-1b**): mp 113-115°C

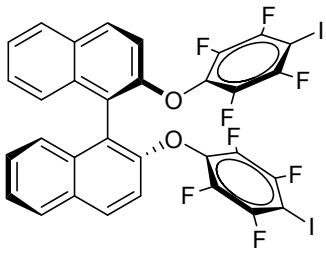
(1*S*,2*S*)-(+)-1,2-di(2,3,5,6-tetrafluoro-4-iodophenoxymethyl)cyclohexane ((*S*,*S*)-2)



Yield: 77%, colourless solid (mp 49-56°C).
$[\alpha]^{22}_{D}$ +18 (c 1.0 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.34-4.25 (m, 4H), 2.02-1.92 (m, 2H), 1.92-1.79 (m, 4H), 1.53-1.25 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.52, 146.08, 142.01, 139.49, 138.42, 78.05, 63.77 (t, J_{CF} = 28.1 Hz), 39.76, 29.44, 25.68. ^{19}F NMR (376 MHz, $CDCl_3$): δ -121.32, -154.53.

Complex with apy (**apy·(*S*,*S*)-2**): mp 153-154°C

(*R*)-(–)-2,2'-bis(2,3,5,6-tetrafluoro-4-iodophenoxy)-1,1'-binaphthyl ((*R*)-3)



Yield: 43%, pale yellow solid (mp 186-189°C).
$[\alpha]^{22}_{D}$ –57.5 (c 0.4 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 9.0 Hz, 2H), 7.86 (d, *J* = 8.1 Hz, 2H), 7.47-7.41 (m, 2H), 7.39 (d, *J* = 9.0, 2H), 7.34-7.29 (m, 2H), 7,18 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.23, 148.01, 145.58, 141.22, 138.71, 135.25, 133.29, 130.55, 130.40, 128.12, 127.20, 125.48, 125.15, 118.78, 117.51, 65.21 (t, J_{CF} = 28.0 Hz). ^{19}F NMR (376 MHz, $CDCl_3$): δ -121.13, -150.87.

Complex with apy (**apy·(*R*)-3**): mp 188-191°C

^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra of donors 1–3

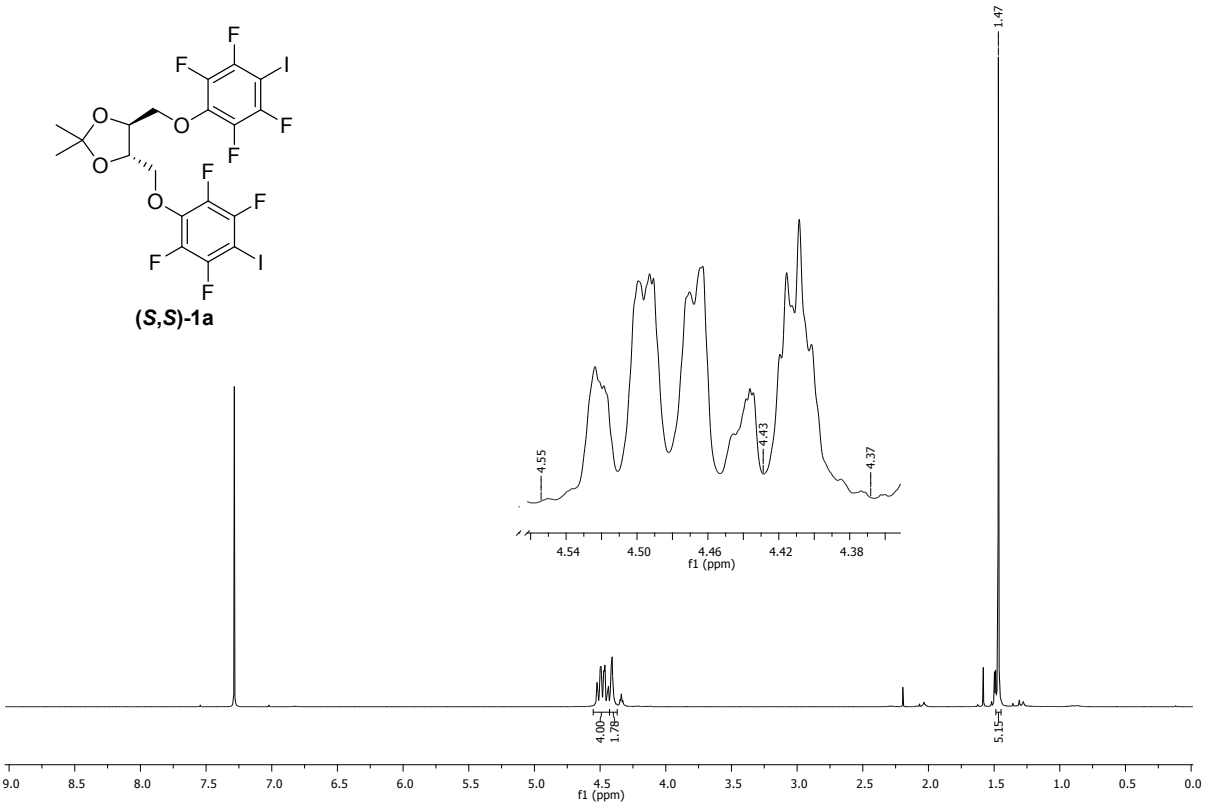


Figure S1. ^{1}H NMR spectrum of **(*S*,*S*)-1a**.

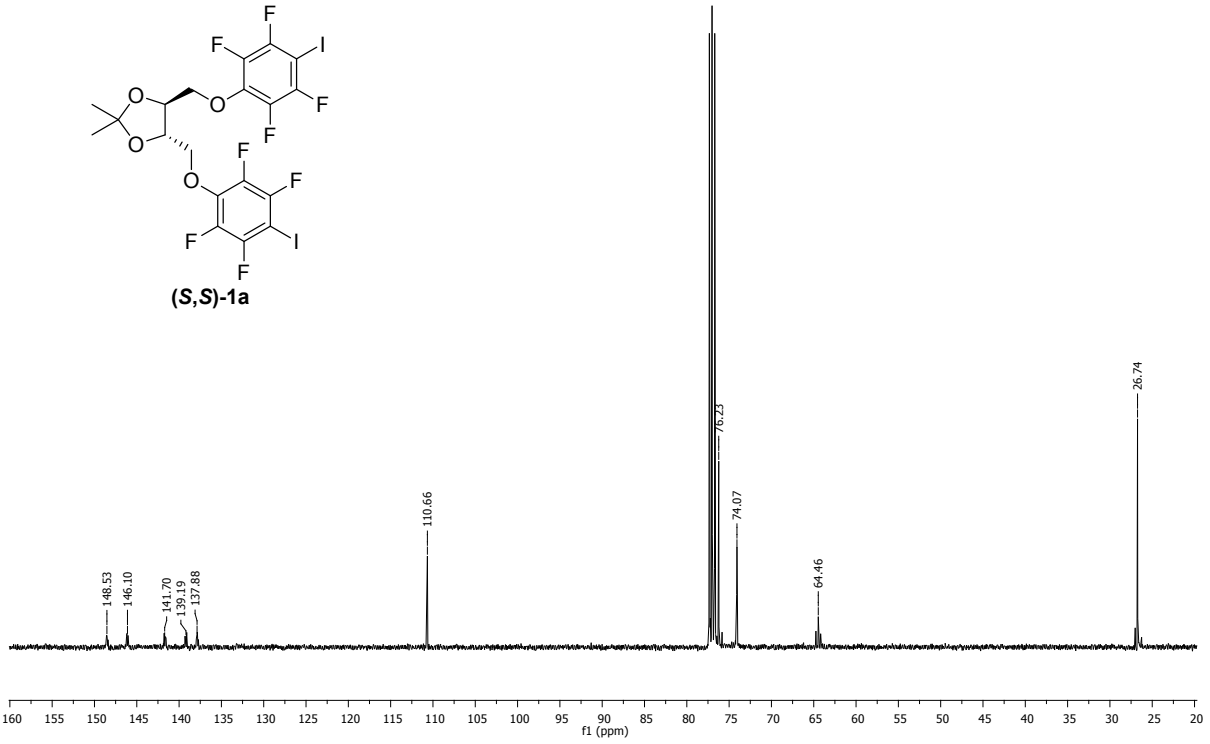


Figure S2. ^{13}C NMR spectrum of **(*S*,*S*)-1a**.

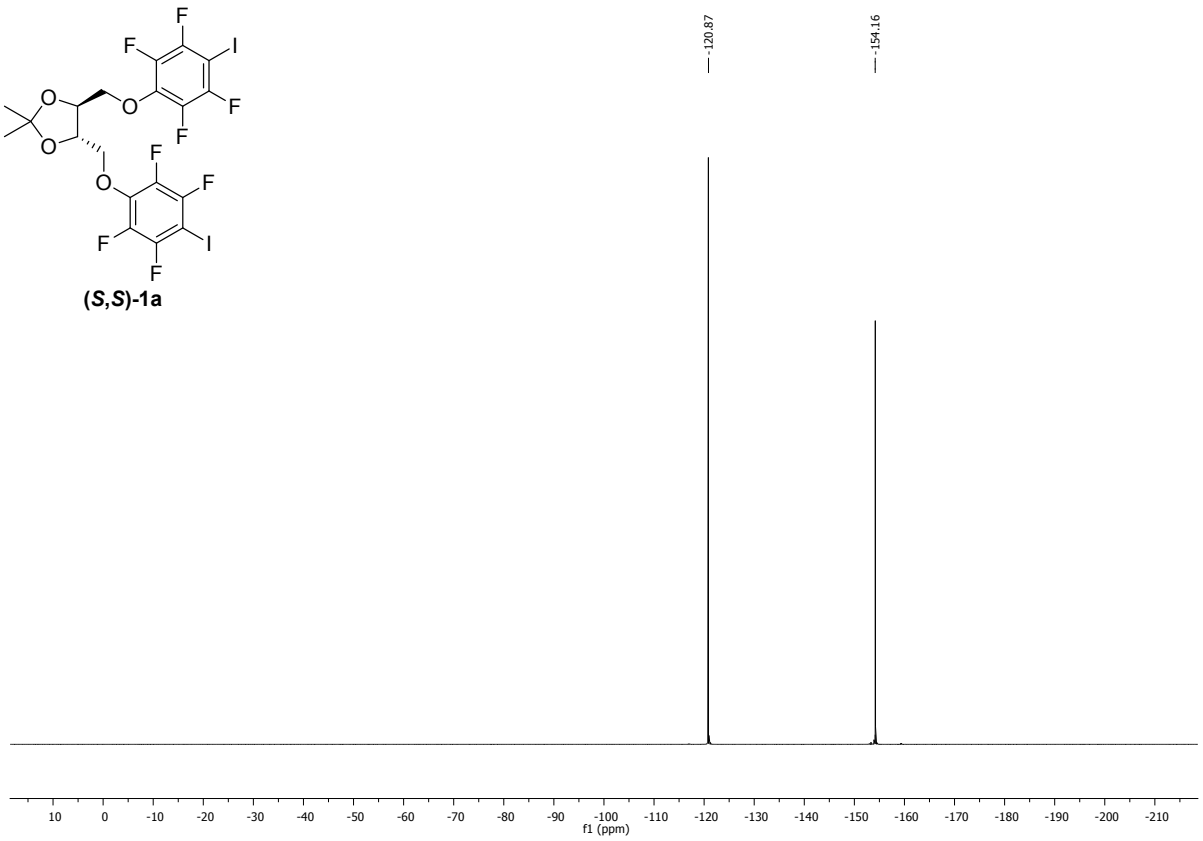


Figure S3. ^{19}F NMR spectrum of **(*S*,*S*)-1a**.

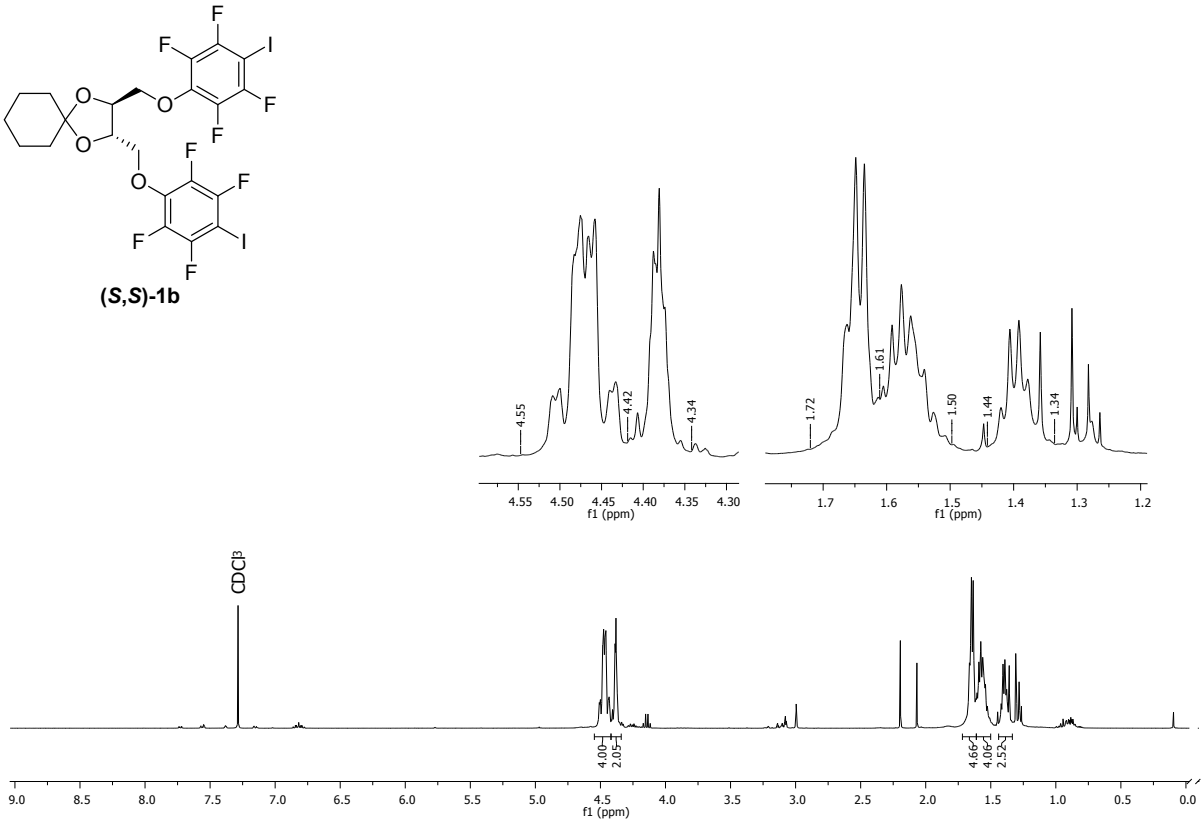


Figure S4. ^{1}H NMR spectrum of **(*S*,*S*)-1b**.

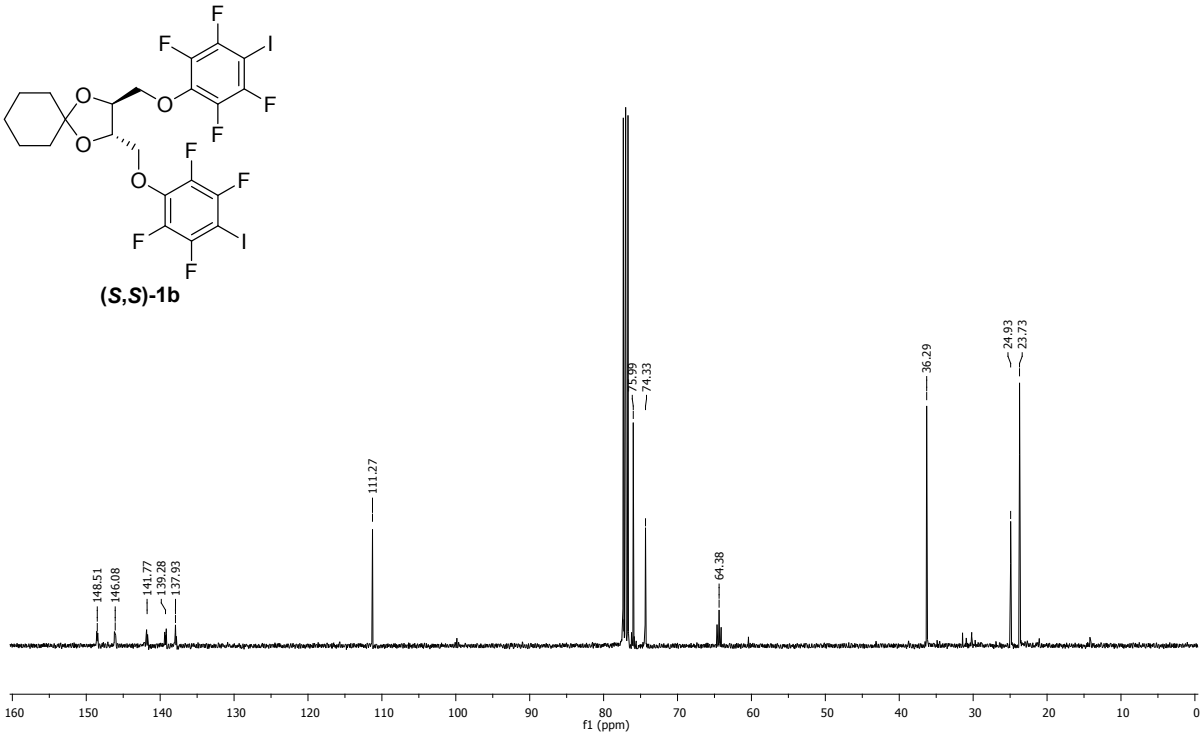


Figure S5. ^{13}C NMR spectrum of **(*S*,*S*)-1b**.

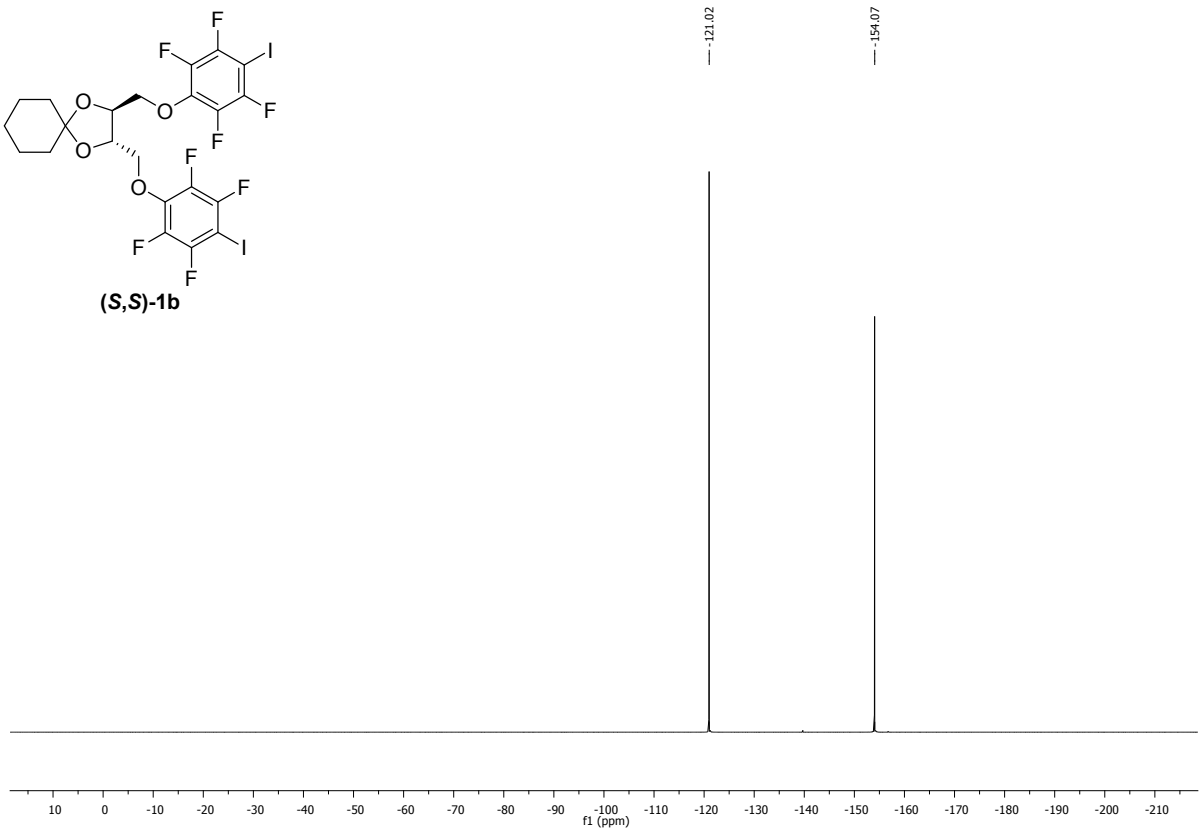


Figure S6. ^{19}F NMR spectrum of **(*S*,*S*)-1b**.

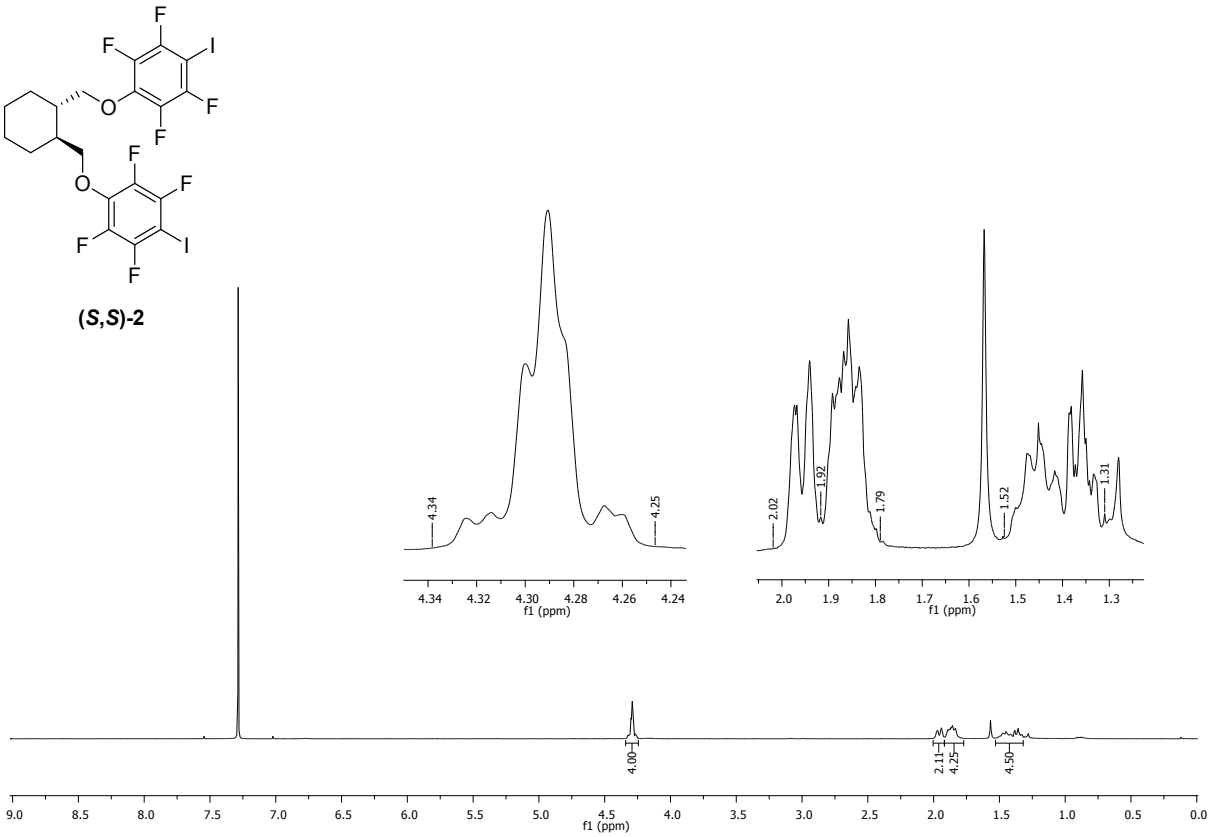


Figure S7. ^{1}H NMR spectrum of **(*S*,*S*)-2**.

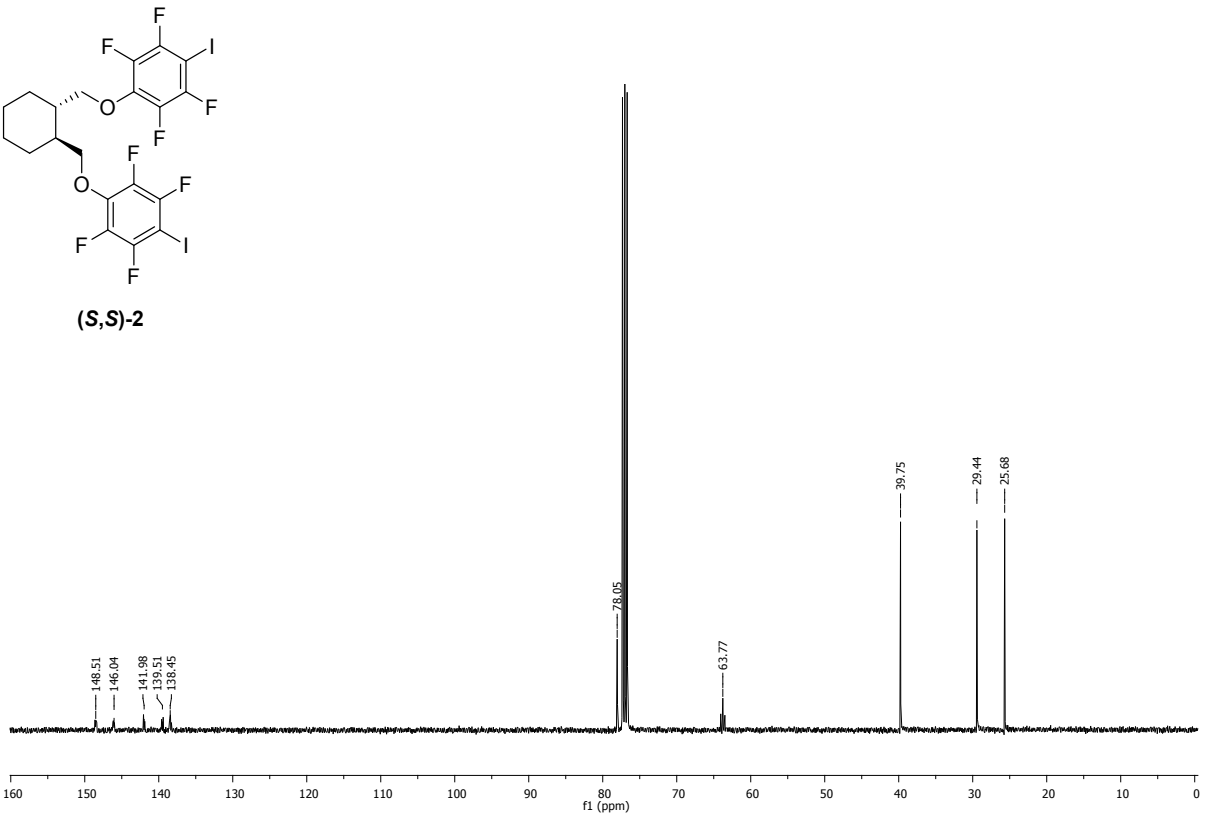


Figure S8. ^{13}C NMR spectrum of **(*S*,*S*)-2**.

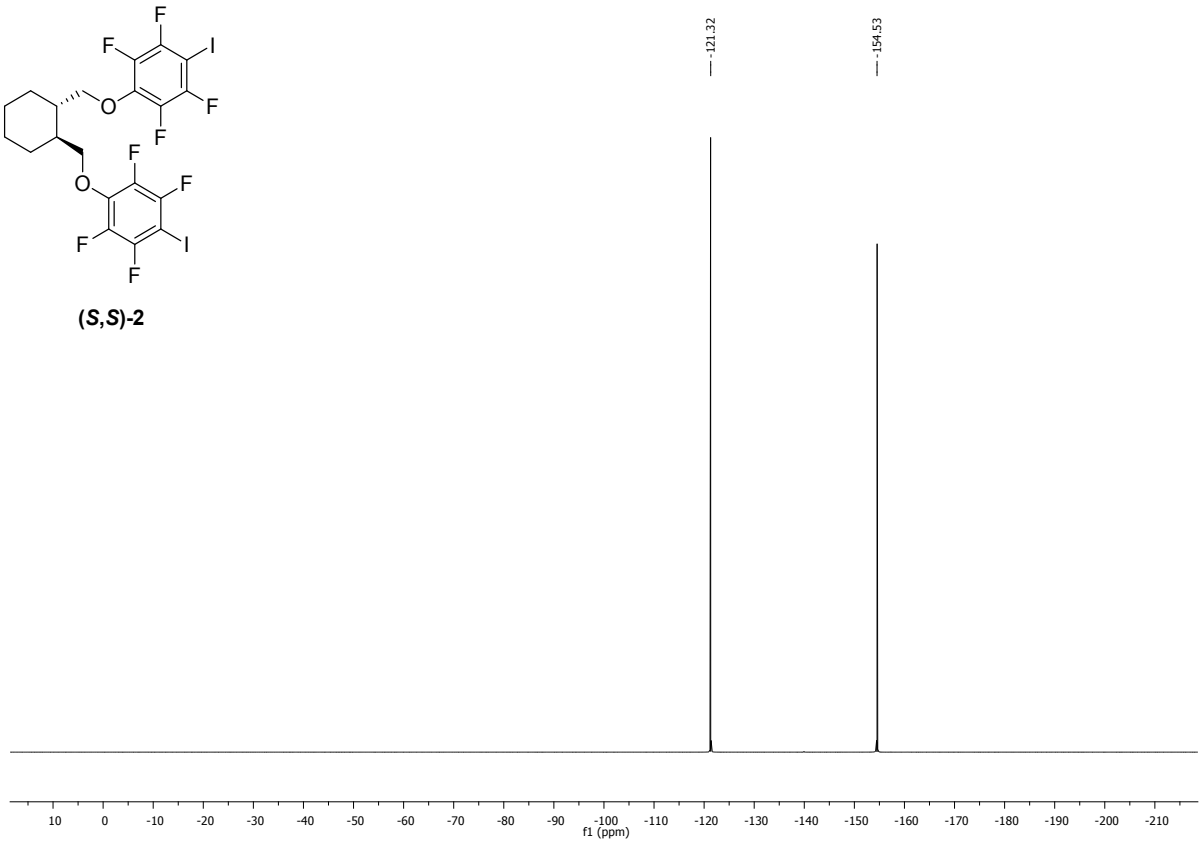


Figure S9. ^{19}F NMR spectrum of **(*S*,*S*)-2**.

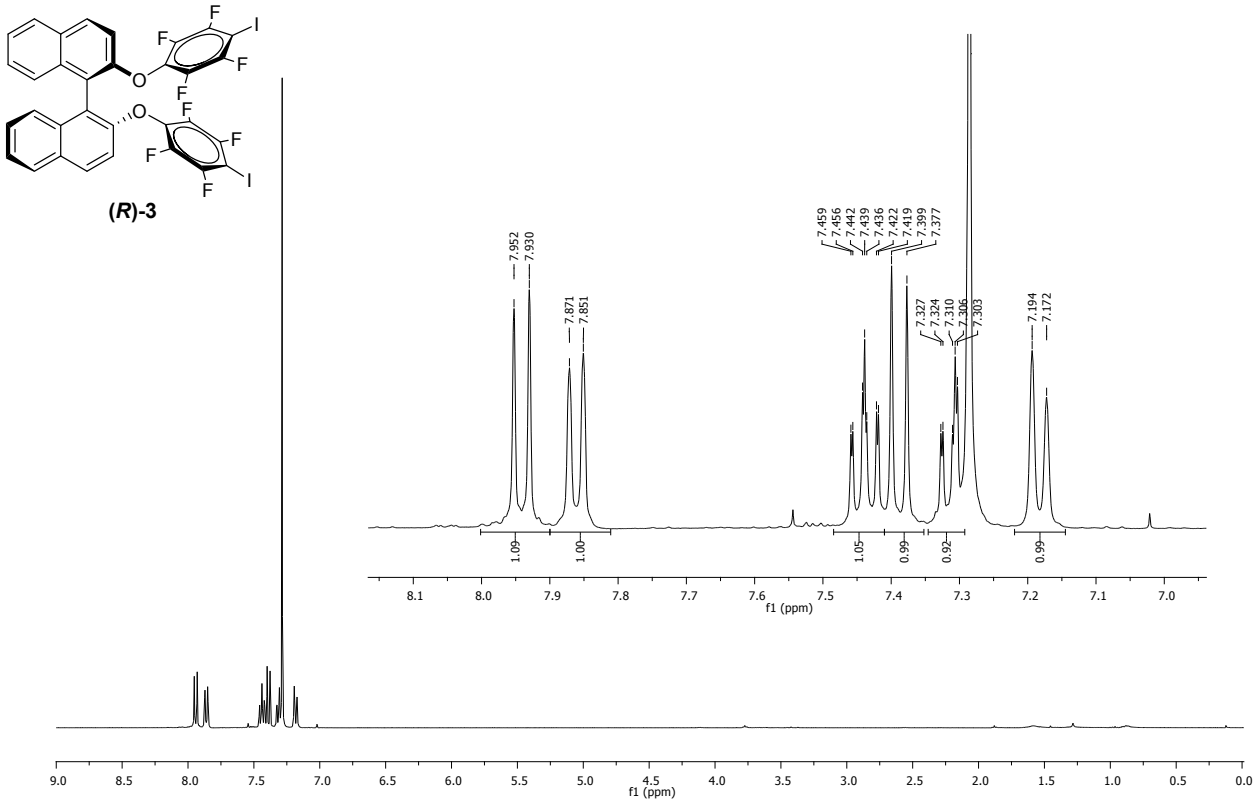


Figure S10. ^{1}H NMR spectrum of (***R***)-**3**.

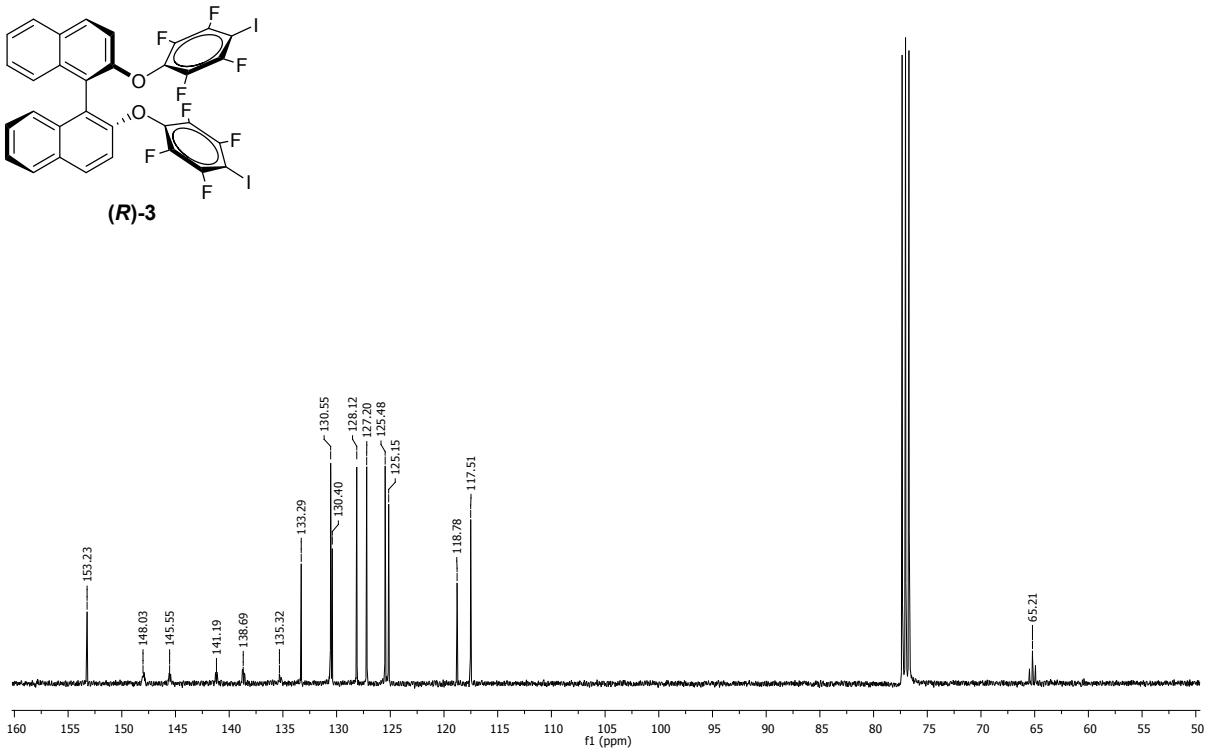


Figure S11. ^{13}C NMR spectrum of (***R***)-**3**.

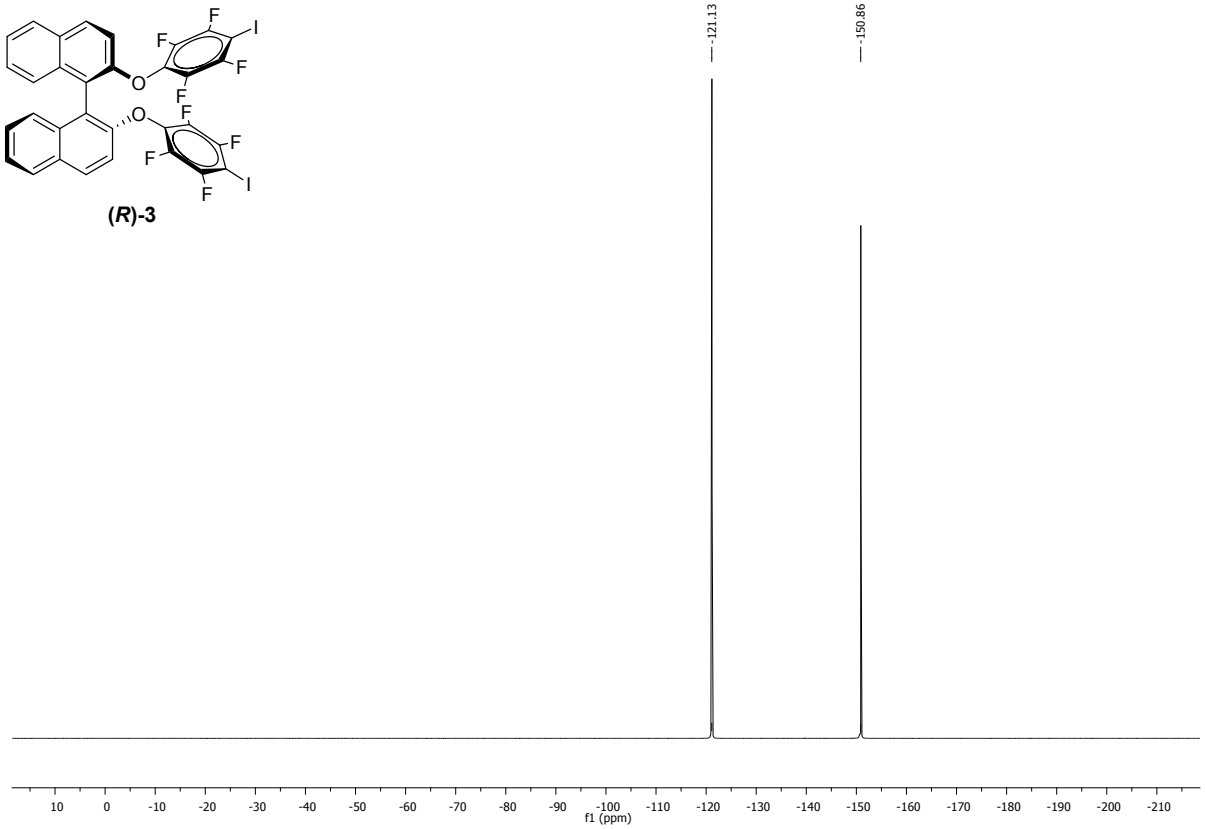


Figure S12. ^{19}F NMR spectrum of (***R***)**-3**.

X-ray crystallography

Table 1. Selected stacking parameters with the shortest distances between centroids [Å], Cg···Cg. Angle α [°] is the dihedral angle between the planes, while CgI_perp is the distance [Å] of the centroid of ring I and square mean plane of ring J. When no symmetry code is given the interaction is intramolecular.

Ring I	Ring J	(symmetry code for ring J)	Cg···Cg	α	CgI_perp
		apy·(*S*,*S*)-1b			
C11-C16	C33-C38	(1+x, y, –1+z)	3.654(5)	0.7(4)	3.323(3)
C1-C6	C43–C48	(–1+x, y, z)	3.874(5)	2.0(4)	3.412(3)
		apy·(*S*,*S*)-2			
C1-C6	N4-C26-C30	(1/2+x, 1/2–y, 1–z)	4.255(3)	4.2(3)	–3.354(2)
C15-C20	N3-C21-C25	(1–x, 1/2+y, 3/2–z)	4.253(3)	5.1(3)	3.335(2)
		apy·(*R*)-3			
C1-C6	C23-C32		3.364(4)	13.4(3)	–3.203(3)
C1-C6	C27-C32		3.565(4)	10.5(3)	–3.243(3)
C7-C12	C13-C22		3.630(4)	18.0(3)	–3.306(3)
C7-C12	C16-C21		3.660(4)	16.8(3)	–3.342(3)
C7-C12	N4-C38-C42	(3/2+x, 1/2–y, –z)	3.681(4)	7.1(3)	–3.391(3)
N3-C33-C37	C16-C21	(–x, 1/2+y, 1/2–z)	3.818(4)	3.1(3)	3.484(3)

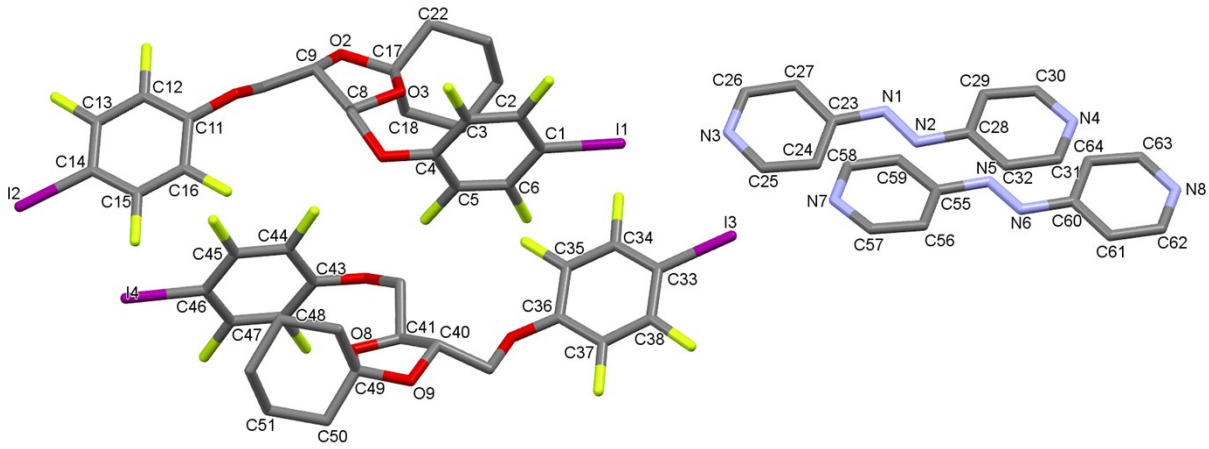


Figure S13. Molecular structure of **apy·(*S*,*S*)-1b**. Hydrogen atoms omitted, atom labelling scheme given for selected atoms of the rings.

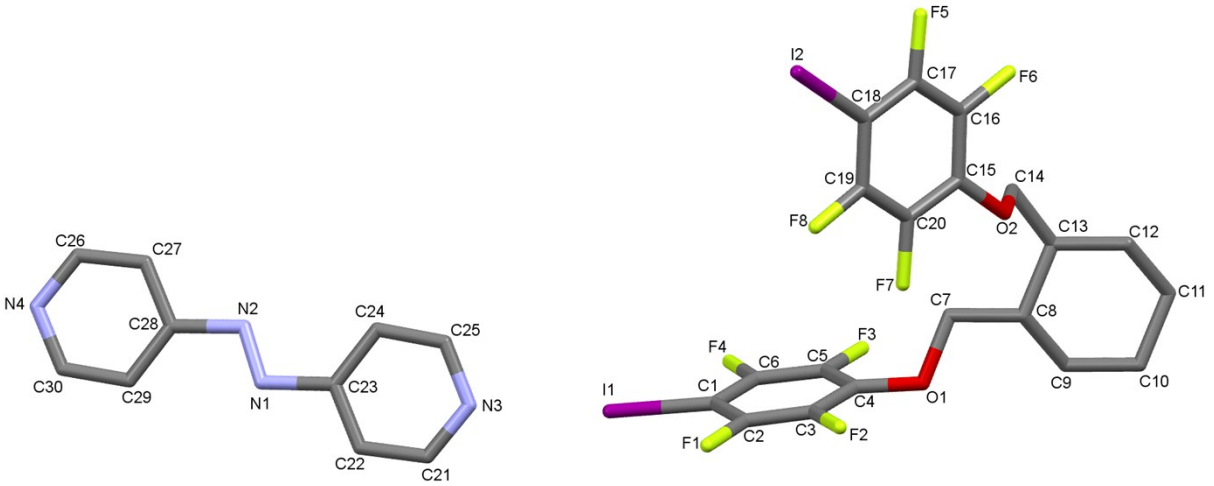


Figure S14. Molecular structure of **apy·(*S*,*S*)-2** and labelling of the atoms. Hydrogen atoms are omitted for clarity.

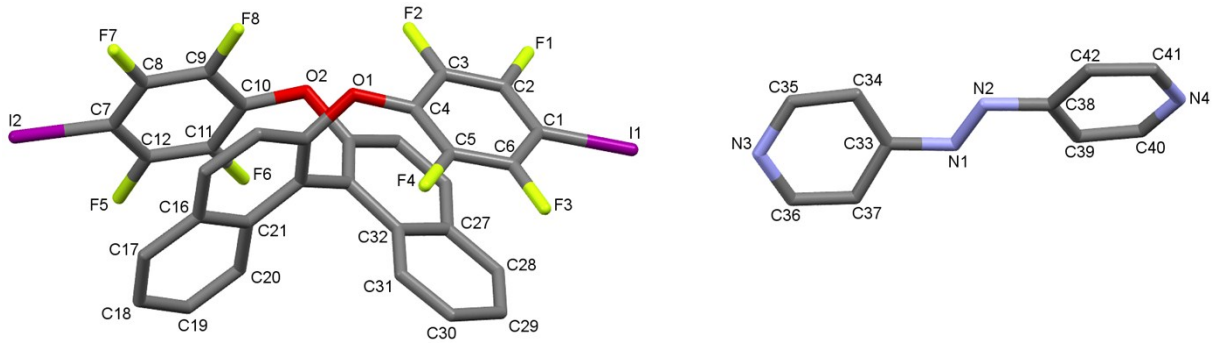


Figure S15. Molecular structure of **apy·(*R*)-3** and labelling of selected atoms. Hydrogen atoms are omitted.

Table 2. Crystal data, data collection and structure refinement details

	apy·(*S*,*S*)-1b	**apy·(*S*,*S*)-2**	**apy·(*R*)-3**
Crystal data			
Chemical formula	$C_{22}H_{16}F_8I_2O_4·C_{10}H_8N_4$	$C_{20}H_{14}F_8I_2O_2·C_{10}H_8N_4$	$C_{32}H_{12}F_8I_2O_2·C_{10}H_8N_4$
M_r	934.35	876.31	1018.42
Crystal system, space group	Triclinic, *P*1	Orthorhombic, $P2_12_12_1$	Orthorhombic, $P2_12_12_1$
Temperature (K)	120	120	120
a, *b*, *c* (Å)	10.4002 (10), 13.3883 (13), 14.2970 (14)	5.2031 (2), 17.8566 (4), 32.8945 (8)	11.5438 (5), 14.8242 (7), 21.8290 (17)
α, *β*, *γ* (°)	68.135 (8), 70.540 (8), 69.104 (7)	90, 90, 90	90, 90, 90
V (Å^3)	1679.3 (3)	3056.22 (15)	3735.5 (4)
Z	2	4	4
Radiation type	Mo *K*α	Mo *K*α	Mo *K*α
μ (mm^{-1})	1.97	2.14	1.77
Crystal size (mm)	0.11×0.17×0.42	0.05×0.07×0.38	0.09×0.18×0.21

Data collection			
Diffractometer	STOE IPDS 2T	STOE IPDS 2T	STOE IPDS 2T
Absorption correction	Numerical	Numerical	Numerical
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	44176, 17217, 16177	38579, 8255, 6979	54653, 10101, 8360
R_{int}	0.054	0.047	0.052
$(\sin\theta / \lambda)_{max}$ (Å^{-1})	0.692	0.688	0.688
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.137, 1.07	0.036, 0.091, 1.08	0.046, 0.123, 1.09
No. of reflections	17211	8255	10101
No. of parameters	901	415	523
No. of restraints	3	0	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rangle_{max}$, $\Delta\rangle_{min}$ (e Å^{-3})	1.56, -1.76	1.32, -0.62	0.96, -1.24
Absolute structure	Classical Flack method preferred over Parsons because s.u. lower.	Flack x determined using 2672 quotients [(I+)-(I-)]/[(I+)+(I-)] [8]	Flack x determined using 3261 quotients [(I+)-(I-)]/[(I+)+(I-)] [8]
Absolute structure parameter	0.05 (2)	-0.018 (15)	-0.030 (14)
CCDC code	1868141	1868142	1868143

Computing details

Program(s) used to solve structure: *SHELXS*[9] for **apy·(*S*,*S*)-1b**, **apy·(*R*)-3**; ShelXT[10] for **apy·(*S*,*S*)-2**. For all compounds, program(s) used to refine structure: *SHELXL*[10]; molecular graphics: Olex2[11]; software used to prepare material for publication: Olex2[11].

Document origin: *publCIF*[12].

apy·(*S*,*S*)-1b

Crystal data

$C_{22}H_{16}F_8I_2O_4 \cdot C_{10}H_8N_4$	$\gamma = 69.104\ (7)°$
$M_r = 934.35$	$V = 1679.3\ (3)$ Å^3
Triclinic, $P\bar{1}$	$Z = 2$
$a = 10.4002\ (10)$ Å	$F(000) = 908$
$b = 13.3883\ (13)$ Å	$D_x = 1.849$ Mg m^{-3}
$c = 14.2970\ (14)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\alpha = 68.135\ (8)°$	$\mu = 1.96$ mm^{-1}
$\beta = 70.540\ (8)°$	$T = 120$ K

Data collection

44176 measured reflections	$\theta_{max} = 29.5°$, $\theta_{min} = 2.2°$
17217 independent reflections	$h = -14 \rightarrow 14$
16177 reflections with $I > 2\sigma(I)$	$k = -17 \rightarrow 18$
$R_{int} = 0.054$	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.0972P)^2 + 1.7781P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.137$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.07$	$\Delta\rangle_{max} = 1.56$ e Å^{-3}
17211 reflections	$\Delta\rangle_{min} = -1.76$ e Å^{-3}
901 parameters	Absolute structure: Classical Flack method preferred over Parsons because s.u. lower.
3 restraints	Absolute structure parameter: 0.05 (2)
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²) for **apy·(*S*,*S*)-1b**

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.01129 (4)	0.39000 (3)	0.42976 (3)	0.02793 (12)
I2	1.60681 (4)	-0.08342 (4)	-0.03516 (3)	0.02979 (12)
F6	1.5100 (5)	0.1860 (5)	-0.1119 (4)	0.0360 (11)
F5	1.2563 (5)	0.3330 (4)	-0.0966 (4)	0.0316 (10)
F8	1.0399 (5)	0.0411 (5)	0.1064 (4)	0.0382 (11)
F7	1.2944 (6)	-0.1088 (5)	0.0901 (5)	0.0414 (12)
F1	0.1232 (5)	0.3869 (4)	0.1926 (4)	0.0320 (10)
F2	0.3714 (5)	0.2904 (4)	0.0846 (3)	0.0305 (9)
F4	0.3104 (6)	0.2352 (5)	0.4917 (3)	0.0367 (11)
F3	0.5614 (5)	0.1525 (4)	0.3795 (4)	0.0340 (10)
O1	1.0106 (6)	0.2618 (5)	0.0097 (4)	0.0291 (11)
O2	0.7754 (6)	0.4743 (5)	0.1307 (5)	0.0302 (11)
O3	0.5957 (5)	0.3903 (5)	0.1884 (4)	0.0271 (10)
O7	0.6019 (5)	0.1729 (4)	0.1748 (4)	0.0252 (10)
C1	0.2078 (7)	0.3169 (7)	0.3454 (6)	0.0236 (13)
C2	0.2289 (7)	0.3282 (6)	0.2414 (5)	0.0220 (12)
C3	0.3576 (7)	0.2794 (6)	0.1850 (5)	0.0215 (12)
C4	0.4731 (7)	0.2196 (6)	0.2293 (5)	0.0215 (12)
C5	0.4514 (8)	0.2074 (6)	0.3337 (6)	0.0240 (13)
C6	0.3231 (8)	0.2518 (7)	0.3904 (5)	0.0252 (13)
C7	0.6801 (7)	0.2520 (6)	0.1002 (5)	0.0255 (12)
H7A	0.621481	0.307514	0.051652	0.031*
H7B	0.766923	0.211814	0.059430	0.031*
C8	0.7202 (7)	0.3117 (6)	0.1533 (5)	0.0216 (12)
H8	0.762170	0.257286	0.212577	0.026*
C9	0.8163 (7)	0.3886 (5)	0.0817 (5)	0.0235 (11)
H9	0.795678	0.421208	0.011253	0.028*
C10	0.9725 (7)	0.3349 (6)	0.0731 (5)	0.0243 (13)
H10A	1.026151	0.392116	0.040137	0.029*
H10B	0.992237	0.292005	0.142395	0.029*
C11	1.1424 (7)	0.1902 (6)	0.0058 (6)	0.0241 (13)
C12	1.2650 (8)	0.2244 (7)	-0.0494 (6)	0.0251 (13)
C13	1.3969 (8)	0.1460 (7)	-0.0572 (6)	0.0268 (14)
C14	1.4099 (7)	0.0351 (7)	-0.0119 (6)	0.0269 (14)
C15	1.2871 (9)	0.0007 (7)	0.0444 (6)	0.0274 (14)
C16	1.1560 (8)	0.0769 (7)	0.0526 (6)	0.0264 (14)

C17	0.6393 (7)	0.4721 (6)	0.2011 (6)	0.0243 (13)
C18	0.6535 (8)	0.4433 (7)	0.3119 (6)	0.0271 (15)
H18A	0.718738	0.368001	0.330688	0.033*
H18B	0.693946	0.497601	0.316316	0.033*
C19	0.5095 (10)	0.4459 (8)	0.3873 (6)	0.0350 (17)
H19A	0.475226	0.384874	0.389246	0.042*
H19B	0.519494	0.432843	0.457733	0.042*
C20	0.4014 (11)	0.5564 (9)	0.3572 (8)	0.044 (2)
H20A	0.429567	0.616294	0.363861	0.053*
H20B	0.308223	0.552617	0.405371	0.053*
C21	0.3880 (10)	0.5853 (9)	0.2470 (8)	0.043 (2)
H21A	0.321537	0.660127	0.228717	0.052*
H21B	0.348915	0.530455	0.242126	0.052*
C22	0.5321 (8)	0.5848 (7)	0.1705 (6)	0.0303 (15)
H22A	0.522004	0.599147	0.099872	0.036*
H22B	0.566609	0.645043	0.169979	0.036*
I3	-0.16451 (4)	0.40044 (4)	0.78940 (3)	0.03087 (12)
I4	1.45020 (4)	-0.06587 (3)	0.32795 (3)	0.02605 (11)
F12	-0.0525 (5)	0.1295 (5)	0.8670 (4)	0.0376 (11)
F11	0.2047 (5)	-0.0088 (4)	0.8458 (4)	0.0335 (10)
F9	0.1407 (6)	0.4352 (4)	0.6620 (4)	0.0396 (11)
F10	0.3982 (5)	0.2957 (4)	0.6384 (4)	0.0356 (11)
F15	1.3181 (5)	-0.0534 (5)	0.5640 (4)	0.0370 (11)
F16	1.0600 (6)	0.0379 (5)	0.6621 (4)	0.0364 (10)
F13	0.9084 (5)	0.1809 (5)	0.3492 (4)	0.0402 (12)
F14	1.1646 (5)	0.0884 (5)	0.2518 (4)	0.0346 (10)
O4	0.4444 (6)	0.0778 (5)	0.7294 (5)	0.0313 (11)
O5	0.8441 (6)	0.1543 (5)	0.5638 (5)	0.0322 (12)
O8	0.8490 (6)	-0.0673 (5)	0.5709 (6)	0.0349 (13)
O9	0.6644 (6)	-0.1422 (5)	0.6144 (4)	0.0276 (10)
C33	0.0349 (8)	0.2893 (7)	0.7633 (6)	0.0251 (13)
C34	0.1518 (9)	0.3267 (8)	0.7071 (6)	0.0299 (16)
C35	0.2862 (8)	0.2551 (7)	0.6940 (6)	0.0256 (14)
C36	0.3081 (7)	0.1392 (7)	0.7392 (6)	0.0243 (13)
C37	0.1896 (8)	0.1001 (6)	0.7969 (6)	0.0255 (13)
C38	0.0552 (8)	0.1738 (7)	0.8077 (5)	0.0258 (13)
C39	0.4842 (8)	-0.0316 (6)	0.7137 (6)	0.0263 (13)
H39A	0.402789	-0.045563	0.704153	0.032*
H39B	0.516319	-0.090822	0.774124	0.032*
C40	0.6034 (7)	-0.0302 (6)	0.6172 (5)	0.0227 (11)

H40	0.567102	0.017512	0.553622	0.027*
C41	0.7289 (7)	0.0032 (6)	0.6202 (5)	0.0235 (12)
H41	0.732095	-0.011732	0.693398	0.028*
C42	0.7253 (7)	0.1238 (6)	0.5602 (6)	0.0311 (14)
H42A	0.727115	0.135991	0.487150	0.037*
H42B	0.635871	0.172804	0.589507	0.037*
C43	0.9737 (7)	0.1074 (6)	0.5103 (6)	0.0248 (13)
C44	1.0073 (8)	0.1208 (7)	0.4047 (6)	0.0267 (14)
C45	1.1413 (8)	0.0726 (6)	0.3540 (6)	0.0244 (13)
C46	1.2494 (7)	0.0109 (6)	0.4049 (6)	0.0210 (12)
C47	1.2173 (7)	0.0026 (6)	0.5092 (6)	0.0251 (13)
C48	1.0841 (8)	0.0497 (6)	0.5606 (6)	0.0256 (13)
C49	0.8068 (8)	-0.1490 (6)	0.5544 (6)	0.0251 (13)
C50	0.8995 (9)	-0.2637 (7)	0.5963 (7)	0.0345 (16)
H50A	0.863640	-0.321415	0.593201	0.041*
H50B	0.896502	-0.275965	0.669575	0.041*
C51	1.0538 (9)	-0.2740 (8)	0.5317 (8)	0.041 (2)
H51A	1.092603	-0.221248	0.540371	0.049*
H51B	1.112658	-0.350607	0.556963	0.049*
C52	1.0601 (10)	-0.2489 (8)	0.4181 (9)	0.045 (2)
H52A	1.029043	-0.305838	0.408995	0.054*
H52B	1.159119	-0.253486	0.378085	0.054*
C53	0.9664 (10)	-0.1330 (8)	0.3759 (7)	0.0392 (19)
H53A	1.002730	-0.075399	0.378792	0.047*
H53B	0.969539	-0.120821	0.302618	0.047*
C54	0.8138 (9)	-0.1214 (8)	0.4391 (7)	0.0327 (17)
H54A	0.773546	-0.172456	0.429086	0.039*
H54B	0.756407	-0.044042	0.414311	0.039*
N1	-0.6571 (7)	0.6577 (6)	0.6815 (6)	0.0318 (14)
N2	-0.7419 (7)	0.6017 (7)	0.7378 (5)	0.0314 (14)
N3	-0.2635 (8)	0.4899 (7)	0.5300 (6)	0.0327 (15)
N4	-1.1277 (8)	0.7731 (7)	0.8964 (6)	0.0377 (17)
C23	-0.5248 (8)	0.5941 (8)	0.6329 (6)	0.0289 (15)
C24	-0.4868 (9)	0.4786 (7)	0.6525 (7)	0.0300 (16)
H24	-0.548654	0.434612	0.699910	0.036*
C25	-0.3537 (8)	0.4312 (8)	0.5992 (7)	0.0335 (16)
H25	-0.324628	0.352396	0.612474	0.040*
C26	-0.3020 (9)	0.5987 (8)	0.5146 (7)	0.0340 (17)
H26	-0.237720	0.640578	0.467315	0.041*
C27	-0.4315 (10)	0.6550 (8)	0.5638 (7)	0.0351 (17)

H27	-0.455567	0.733720	0.550381	0.042*
C28	-0.8714 (8)	0.6653 (7)	0.7887 (6)	0.0273 (14)
C29	-0.8989 (8)	0.7763 (7)	0.7806 (6)	0.0323 (16)
H29	-0.831332	0.817400	0.739297	0.039*
C30	-1.0298 (9)	0.8258 (8)	0.8355 (7)	0.0365 (18)
H30	-1.050345	0.902922	0.828848	0.044*
C31	-1.0977 (9)	0.6639 (8)	0.9034 (7)	0.0365 (18)
H31	-1.166070	0.624210	0.946507	0.044*
C32	-0.9713 (9)	0.6072 (8)	0.8503 (6)	0.0324 (16)
H32	-0.954021	0.530678	0.856175	0.039*
N5	-0.8129 (7)	0.7170 (6)	1.0158 (6)	0.0291 (13)
N6	-0.9018 (7)	0.6637 (6)	1.0645 (6)	0.0325 (14)
N7	-0.4327 (7)	0.5456 (7)	0.8521 (6)	0.0323 (15)
N8	-1.2824 (7)	0.8357 (6)	1.2260 (6)	0.0311 (14)
C55	-0.6847 (8)	0.6524 (7)	0.9618 (6)	0.0298 (15)
C56	-0.6630 (8)	0.5443 (7)	0.9630 (6)	0.0295 (15)
H56	-0.733737	0.505421	1.000746	0.035*
C57	-0.5344 (8)	0.4944 (7)	0.9073 (6)	0.0299 (15)
H57	-0.517908	0.419412	0.908602	0.036*
C58	-0.4551 (9)	0.6506 (9)	0.8508 (8)	0.041 (2)
H58	-0.382910	0.687602	0.811340	0.049*
C59	-0.5801 (10)	0.7082 (8)	0.9049 (8)	0.0369 (18)
H59	-0.593465	0.782889	0.903010	0.044*
C60	-1.0295 (8)	0.7262 (7)	1.1181 (7)	0.0284 (14)
C61	-1.1268 (9)	0.6669 (7)	1.1842 (7)	0.0338 (17)
H61	-1.109684	0.589811	1.192634	0.041*
C62	-1.2523 (9)	0.7266 (8)	1.2383 (7)	0.0337 (17)
H62	-1.318965	0.687157	1.285969	0.040*
C63	-1.1888 (9)	0.8905 (7)	1.1612 (8)	0.0350 (17)
H63	-1.210488	0.968185	1.152327	0.042*
C64	-1.0608 (9)	0.8399 (8)	1.1057 (8)	0.0346 (18)
H64	-0.995635	0.881797	1.060166	0.041*

Atomic displacement parameters (Å²) for **apy·(*S*,*S*)-1b**

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0216 (2)	0.0297 (2)	0.0343 (2)	-0.00694 (17)	0.00080 (17)	-0.01743 (19)
I2	0.0240 (2)	0.0324 (3)	0.0283 (2)	0.00240 (18)	-0.00785 (17)	-0.01141 (19)
F6	0.024 (2)	0.040 (3)	0.041 (3)	-0.009 (2)	0.0007 (19)	-0.014 (2)

F5	0.031 (2)	0.027 (2)	0.035 (2)	-0.0056 (19)	-0.0057 (19)	-0.0107 (19)
F8	0.027 (2)	0.042 (3)	0.042 (3)	-0.012 (2)	0.0013 (19)	-0.013 (2)
F7	0.040 (3)	0.028 (2)	0.048 (3)	-0.008 (2)	-0.004 (2)	-0.009 (2)
F1	0.026 (2)	0.036 (2)	0.037 (2)	-0.0022 (18)	-0.0174 (18)	-0.010 (2)
F2	0.034 (2)	0.042 (3)	0.0207 (19)	-0.0091 (19)	-0.0074 (16)	-0.0148 (18)
F4	0.037 (2)	0.053 (3)	0.0183 (19)	-0.011 (2)	-0.0033 (17)	-0.0112 (19)
F3	0.027 (2)	0.039 (2)	0.030 (2)	-0.0007 (18)	-0.0142 (17)	-0.0041 (18)
O1	0.022 (2)	0.040 (3)	0.032 (2)	0.000 (2)	-0.0078 (19)	-0.023 (2)
O2	0.028 (3)	0.028 (3)	0.036 (3)	-0.009 (2)	0.003 (2)	-0.019 (2)
O3	0.022 (2)	0.029 (3)	0.033 (3)	-0.005 (2)	0.0030 (19)	-0.021 (2)
O7	0.021 (2)	0.019 (2)	0.031 (2)	-0.0051 (17)	0.0026 (18)	-0.0106 (18)
C1	0.019 (3)	0.029 (3)	0.025 (3)	-0.006 (3)	0.001 (2)	-0.015 (3)
C2	0.023 (3)	0.021 (3)	0.026 (3)	-0.003 (2)	-0.010 (2)	-0.009 (2)
C3	0.023 (3)	0.026 (3)	0.019 (3)	-0.009 (2)	-0.005 (2)	-0.008 (2)
C4	0.021 (3)	0.018 (3)	0.025 (3)	-0.004 (2)	-0.003 (2)	-0.008 (2)
C5	0.022 (3)	0.023 (3)	0.028 (3)	-0.004 (2)	-0.010 (2)	-0.006 (2)
C6	0.031 (3)	0.029 (4)	0.018 (3)	-0.010 (3)	-0.004 (2)	-0.009 (2)
C7	0.023 (3)	0.029 (3)	0.022 (3)	-0.008 (2)	0.001 (2)	-0.011 (2)
C8	0.020 (3)	0.023 (3)	0.021 (3)	-0.004 (2)	0.000 (2)	-0.011 (2)
C9	0.023 (3)	0.022 (3)	0.025 (3)	-0.004 (2)	-0.002 (2)	-0.012 (2)
C10	0.022 (3)	0.029 (3)	0.021 (3)	-0.006 (2)	-0.003 (2)	-0.008 (3)
C11	0.021 (3)	0.029 (3)	0.026 (3)	-0.001 (3)	-0.006 (2)	-0.016 (3)
C12	0.025 (3)	0.028 (3)	0.023 (3)	-0.002 (3)	-0.007 (2)	-0.010 (3)
C13	0.022 (3)	0.033 (4)	0.027 (3)	-0.007 (3)	-0.004 (3)	-0.012 (3)
C14	0.018 (3)	0.033 (4)	0.024 (3)	0.003 (3)	-0.003 (2)	-0.014 (3)
C15	0.028 (3)	0.024 (3)	0.030 (4)	-0.006 (3)	-0.004 (3)	-0.011 (3)
C16	0.023 (3)	0.033 (4)	0.024 (3)	-0.008 (3)	-0.003 (2)	-0.012 (3)
C17	0.022 (3)	0.019 (3)	0.030 (3)	-0.002 (2)	-0.003 (3)	-0.011 (3)
C18	0.027 (3)	0.023 (3)	0.027 (3)	-0.003 (3)	-0.006 (3)	-0.007 (3)
C19	0.040 (4)	0.036 (4)	0.026 (3)	-0.012 (3)	0.002 (3)	-0.012 (3)
C20	0.040 (4)	0.045 (5)	0.043 (5)	-0.010 (4)	0.013 (4)	-0.028 (4)
C21	0.028 (4)	0.038 (5)	0.056 (6)	0.004 (3)	-0.010 (4)	-0.018 (4)
C22	0.032 (4)	0.024 (3)	0.030 (3)	-0.001 (3)	-0.012 (3)	-0.005 (3)
I3	0.0233 (2)	0.0371 (3)	0.0271 (2)	0.00025 (18)	-0.00615 (17)	-0.01127 (19)
I4	0.01985 (19)	0.0249 (2)	0.0323 (2)	-0.00377 (15)	-0.00209 (16)	-0.01255 (17)
F12	0.025 (2)	0.037 (3)	0.044 (3)	-0.012 (2)	0.0011 (19)	-0.008 (2)
F11	0.030 (2)	0.028 (2)	0.033 (2)	-0.0082 (19)	0.0007 (18)	-0.0051 (19)
F9	0.038 (3)	0.028 (2)	0.043 (3)	-0.007 (2)	-0.004 (2)	-0.007 (2)
F10	0.029 (2)	0.036 (2)	0.040 (3)	-0.0156 (19)	0.0062 (19)	-0.015 (2)
F15	0.028 (2)	0.049 (3)	0.033 (2)	-0.001 (2)	-0.0163 (19)	-0.012 (2)

F16	0.043 (3)	0.045 (3)	0.022 (2)	-0.012 (2)	-0.0031 (18)	-0.0130 (19)
F13	0.027 (2)	0.044 (3)	0.043 (3)	0.008 (2)	-0.019 (2)	-0.012 (2)
F14	0.037 (2)	0.042 (3)	0.023 (2)	-0.003 (2)	-0.0071 (18)	-0.0132 (19)
O4	0.022 (2)	0.033 (3)	0.043 (3)	-0.006 (2)	-0.001 (2)	-0.022 (2)
O5	0.022 (2)	0.032 (3)	0.046 (3)	-0.009 (2)	0.002 (2)	-0.022 (2)
O8	0.021 (2)	0.032 (3)	0.056 (4)	-0.007 (2)	0.001 (2)	-0.026 (3)
O9	0.024 (2)	0.027 (3)	0.033 (3)	-0.0081 (19)	-0.001 (2)	-0.014 (2)
C33	0.025 (3)	0.028 (3)	0.022 (3)	-0.003 (3)	-0.004 (3)	-0.011 (3)
C34	0.029 (4)	0.034 (4)	0.025 (3)	-0.003 (3)	-0.003 (3)	-0.014 (3)
C35	0.023 (3)	0.031 (4)	0.023 (3)	-0.009 (3)	0.002 (2)	-0.013 (3)
C36	0.024 (3)	0.030 (3)	0.023 (3)	-0.009 (3)	-0.001 (2)	-0.015 (3)
C37	0.027 (3)	0.025 (3)	0.023 (3)	-0.009 (3)	-0.003 (2)	-0.007 (3)
C38	0.024 (3)	0.032 (4)	0.022 (3)	-0.008 (3)	0.000 (2)	-0.013 (3)
C39	0.029 (3)	0.025 (3)	0.024 (3)	-0.007 (2)	-0.002 (3)	-0.010 (3)
C40	0.022 (3)	0.025 (3)	0.020 (3)	-0.005 (2)	-0.004 (2)	-0.008 (2)
C41	0.019 (3)	0.026 (3)	0.025 (3)	-0.003 (2)	-0.003 (2)	-0.012 (2)
C42	0.018 (3)	0.028 (3)	0.045 (4)	-0.006 (2)	-0.003 (3)	-0.011 (3)
C43	0.020 (3)	0.022 (3)	0.035 (4)	-0.005 (2)	-0.003 (3)	-0.015 (3)
C44	0.023 (3)	0.027 (3)	0.029 (3)	0.002 (3)	-0.012 (3)	-0.009 (3)
C45	0.023 (3)	0.027 (3)	0.022 (3)	-0.004 (3)	-0.008 (2)	-0.006 (2)
C46	0.018 (3)	0.017 (3)	0.028 (3)	-0.003 (2)	-0.007 (2)	-0.007 (2)
C47	0.022 (3)	0.027 (3)	0.028 (3)	-0.004 (3)	-0.009 (3)	-0.010 (3)
C48	0.027 (3)	0.028 (3)	0.023 (3)	-0.009 (3)	-0.004 (2)	-0.010 (3)
C49	0.024 (3)	0.025 (3)	0.027 (3)	-0.006 (3)	-0.005 (3)	-0.009 (3)
C50	0.032 (4)	0.026 (4)	0.041 (4)	-0.006 (3)	-0.010 (3)	-0.006 (3)
C51	0.026 (4)	0.029 (4)	0.056 (5)	0.004 (3)	-0.005 (3)	-0.014 (4)
C52	0.034 (4)	0.034 (4)	0.058 (6)	0.002 (3)	-0.001 (4)	-0.024 (4)
C53	0.037 (4)	0.038 (4)	0.035 (4)	-0.004 (3)	0.001 (3)	-0.016 (3)
C54	0.030 (4)	0.036 (4)	0.032 (4)	-0.005 (3)	-0.008 (3)	-0.013 (3)
N1	0.030 (3)	0.027 (3)	0.031 (3)	-0.002 (3)	-0.002 (3)	-0.010 (3)
N2	0.023 (3)	0.039 (4)	0.029 (3)	-0.003 (3)	-0.003 (2)	-0.014 (3)
N3	0.026 (3)	0.042 (4)	0.036 (4)	-0.007 (3)	-0.003 (3)	-0.024 (3)
N4	0.028 (3)	0.039 (4)	0.031 (4)	-0.002 (3)	-0.004 (3)	-0.002 (3)
C23	0.024 (3)	0.038 (4)	0.023 (3)	-0.006 (3)	-0.001 (3)	-0.013 (3)
C24	0.029 (3)	0.025 (4)	0.037 (4)	-0.005 (3)	-0.002 (3)	-0.019 (3)
C25	0.026 (3)	0.036 (4)	0.043 (4)	-0.004 (3)	-0.003 (3)	-0.024 (4)
C26	0.027 (3)	0.040 (5)	0.036 (4)	-0.009 (3)	0.004 (3)	-0.022 (4)
C27	0.035 (4)	0.031 (4)	0.038 (4)	-0.009 (3)	0.000 (3)	-0.017 (3)
C28	0.022 (3)	0.025 (3)	0.025 (3)	0.001 (3)	-0.004 (3)	-0.005 (3)
C29	0.026 (3)	0.031 (4)	0.031 (4)	0.000 (3)	-0.003 (3)	-0.009 (3)

C30	0.032 (4)	0.029 (4)	0.035 (4)	0.000 (3)	-0.004 (3)	-0.006 (3)
C31	0.025 (3)	0.036 (4)	0.035 (4)	-0.002 (3)	-0.003 (3)	-0.004 (3)
C32	0.026 (3)	0.030 (4)	0.031 (4)	0.001 (3)	-0.003 (3)	-0.009 (3)
N5	0.023 (3)	0.026 (3)	0.034 (3)	-0.001 (2)	-0.004 (2)	-0.012 (3)
N6	0.025 (3)	0.035 (3)	0.034 (3)	-0.006 (3)	0.002 (3)	-0.016 (3)
N7	0.022 (3)	0.036 (4)	0.030 (3)	0.006 (3)	-0.002 (2)	-0.016 (3)
N8	0.020 (3)	0.035 (4)	0.039 (4)	-0.003 (3)	-0.002 (3)	-0.020 (3)
C55	0.024 (3)	0.033 (4)	0.025 (3)	-0.004 (3)	-0.004 (3)	-0.007 (3)
C56	0.022 (3)	0.029 (4)	0.028 (3)	-0.003 (3)	-0.002 (3)	-0.006 (3)
C57	0.028 (3)	0.027 (3)	0.029 (3)	0.003 (3)	-0.006 (3)	-0.011 (3)
C58	0.026 (4)	0.041 (5)	0.040 (5)	-0.005 (3)	0.001 (3)	-0.006 (4)
C59	0.031 (4)	0.028 (4)	0.042 (5)	-0.007 (3)	0.000 (3)	-0.007 (3)
C60	0.024 (3)	0.026 (4)	0.036 (4)	-0.003 (3)	-0.002 (3)	-0.017 (3)
C61	0.028 (4)	0.026 (4)	0.041 (4)	-0.005 (3)	0.005 (3)	-0.015 (3)
C62	0.028 (3)	0.033 (4)	0.035 (4)	-0.008 (3)	0.004 (3)	-0.015 (3)
C63	0.031 (4)	0.022 (4)	0.053 (5)	-0.003 (3)	-0.006 (3)	-0.020 (4)
C64	0.028 (4)	0.039 (5)	0.041 (4)	-0.013 (3)	0.003 (3)	-0.020 (4)

Geometric parameters (Å, °) for **apy·(*S*,*S*)-1b**

I1—C1	2.097 (7)	O8—C49	1.431 (9)
I2—C14	2.108 (7)	O9—C40	1.414 (9)
F6—C13	1.344 (9)	O9—C49	1.432 (9)
F5—C12	1.339 (9)	C33—C34	1.363 (11)
F8—C16	1.341 (9)	C33—C38	1.401 (11)
F7—C15	1.349 (10)	C34—C35	1.382 (11)
F1—C2	1.341 (7)	C35—C36	1.407 (11)
F2—C3	1.350 (7)	C36—C37	1.391 (10)
F4—C6	1.348 (8)	C37—C38	1.391 (10)
F3—C5	1.348 (8)	C39—C40	1.516 (9)
O1—C10	1.443 (9)	C40—C41	1.541 (9)
O1—C11	1.361 (8)	C41—C42	1.514 (10)
O2—C9	1.428 (8)	C43—C44	1.387 (11)
O2—C17	1.441 (9)	C43—C48	1.392 (10)
O3—C8	1.423 (8)	C44—C45	1.385 (11)
O3—C17	1.412 (9)	C45—C46	1.382 (9)
O7—C4	1.365 (8)	C46—C47	1.385 (10)
O7—C7	1.454 (8)	C47—C48	1.378 (10)
C1—C2	1.386 (10)	C49—C50	1.513 (11)
C1—C6	1.401 (10)	C49—C54	1.532 (11)

C2—C3	1.385 (10)	C50—C51	1.548 (12)
C3—C4	1.389 (9)	C51—C52	1.513 (16)
C4—C5	1.387 (10)	C52—C53	1.530 (13)
C5—C6	1.365 (11)	C53—C54	1.527 (13)
C7—C8	1.513 (10)	N1—N2	1.240 (11)
C8—C9	1.541 (9)	N1—C23	1.439 (10)
C9—C10	1.508 (9)	N2—C28	1.435 (10)
C11—C12	1.392 (10)	N3—C25	1.343 (12)
C11—C16	1.386 (11)	N3—C26	1.316 (12)
C12—C13	1.397 (10)	N4—C30	1.326 (12)
C13—C14	1.358 (12)	N4—C31	1.353 (13)
C14—C15	1.396 (11)	C23—C24	1.392 (12)
C15—C16	1.380 (11)	C23—C27	1.382 (12)
C17—C18	1.529 (11)	C24—C25	1.390 (11)
C17—C22	1.530 (10)	C26—C27	1.385 (12)
C18—C19	1.525 (11)	C28—C29	1.376 (12)
C19—C20	1.517 (14)	C28—C32	1.385 (12)
C20—C21	1.518 (15)	C29—C30	1.394 (12)
C21—C22	1.533 (12)	C31—C32	1.390 (12)
I3—C33	2.086 (7)	N5—N6	1.233 (10)
I4—C46	2.100 (7)	N5—C55	1.453 (10)
F12—C38	1.339 (8)	N6—C60	1.434 (10)
F11—C37	1.338 (9)	N7—C57	1.331 (11)
F9—C34	1.331 (11)	N7—C58	1.334 (13)
F10—C35	1.340 (8)	N8—C62	1.334 (12)
F15—C47	1.351 (8)	N8—C63	1.322 (12)
F16—C48	1.345 (8)	C55—C56	1.377 (12)
F13—C44	1.347 (8)	C55—C59	1.393 (12)
F14—C45	1.345 (8)	C56—C57	1.386 (10)
O4—C36	1.352 (9)	C58—C59	1.394 (13)
O4—C39	1.455 (9)	C60—C61	1.387 (11)
O5—C42	1.454 (9)	C60—C64	1.393 (12)
O5—C43	1.365 (9)	C61—C62	1.408 (11)
O8—C41	1.419 (9)	C63—C64	1.384 (12)
C11—O1—C10	115.4 (5)	C36—C37—C38	120.6 (7)
C9—O2—C17	108.4 (5)	F12—C38—C33	121.7 (7)
C17—O3—C8	107.3 (5)	F12—C38—C37	116.7 (7)
C4—O7—C7	115.0 (5)	C37—C38—C33	121.5 (7)
C2—C1—I1	121.1 (5)	O4—C39—C40	106.3 (6)

C2—C1—C6	116.2 (6)	O9—C40—C39	106.7 (5)
C6—C1—I1	122.7 (5)	O9—C40—C41	102.7 (5)
F1—C2—C1	120.2 (6)	C39—C40—C41	114.3 (6)
F1—C2—C3	118.0 (6)	O8—C41—C40	103.7 (6)
C3—C2—C1	121.8 (6)	O8—C41—C42	109.2 (6)
F2—C3—C2	119.4 (6)	C42—C41—C40	112.5 (6)
F2—C3—C4	119.2 (6)	O5—C42—C41	111.9 (6)
C2—C3—C4	121.4 (6)	O5—C43—C44	123.9 (7)
O7—C4—C3	122.6 (6)	O5—C43—C48	119.5 (7)
O7—C4—C5	120.7 (6)	C44—C43—C48	116.3 (6)
C5—C4—C3	116.7 (6)	F13—C44—C43	120.1 (7)
F3—C5—C4	118.9 (7)	F13—C44—C45	118.4 (7)
F3—C5—C6	119.2 (7)	C45—C44—C43	121.5 (7)
C6—C5—C4	121.8 (7)	F14—C45—C44	117.7 (6)
F4—C6—C1	119.8 (7)	F14—C45—C46	120.2 (7)
F4—C6—C5	118.2 (7)	C46—C45—C44	122.1 (7)
C5—C6—C1	121.9 (7)	C45—C46—I4	121.6 (5)
O7—C7—C8	111.4 (5)	C45—C46—C47	116.2 (6)
O3—C8—C7	107.9 (5)	C47—C46—I4	122.2 (5)
O3—C8—C9	101.2 (5)	F15—C47—C46	119.9 (6)
C7—C8—C9	115.3 (5)	F15—C47—C48	117.8 (7)
O2—C9—C8	103.2 (5)	C48—C47—C46	122.2 (6)
O2—C9—C10	108.1 (6)	F16—C48—C43	119.3 (7)
C10—C9—C8	115.0 (6)	F16—C48—C47	119.1 (7)
O1—C10—C9	105.1 (5)	C47—C48—C43	121.5 (7)
O1—C11—C12	122.9 (7)	O8—C49—O9	105.4 (6)
O1—C11—C16	119.1 (7)	O8—C49—C50	109.1 (6)
C16—C11—C12	117.9 (7)	O8—C49—C54	109.8 (7)
F5—C12—C11	119.7 (7)	O9—C49—C50	108.9 (6)
F5—C12—C13	119.9 (7)	O9—C49—C54	111.0 (6)
C11—C12—C13	120.4 (7)	C50—C49—C54	112.5 (7)
F6—C13—C12	116.7 (7)	C49—C50—C51	110.0 (7)
F6—C13—C14	121.7 (7)	C52—C51—C50	110.7 (8)
C14—C13—C12	121.6 (7)	C51—C52—C53	111.7 (8)
C13—C14—I2	121.3 (5)	C54—C53—C52	110.6 (8)
C13—C14—C15	118.1 (7)	C53—C54—C49	110.8 (7)
C15—C14—I2	120.5 (6)	N2—N1—C23	113.9 (7)
F7—C15—C14	120.3 (7)	N1—N2—C28	113.3 (7)
F7—C15—C16	118.6 (7)	C26—N3—C25	117.8 (7)
C16—C15—C14	121.1 (8)	C30—N4—C31	116.8 (8)

F8—C16—C11	119.4 (7)	C24—C23—N1	124.5 (7)
F8—C16—C15	119.7 (7)	C27—C23—N1	115.5 (8)
C15—C16—C11	120.9 (7)	C27—C23—C24	119.9 (8)
O2—C17—C18	109.4 (6)	C25—C24—C23	116.5 (8)
O2—C17—C22	109.8 (6)	N3—C25—C24	124.1 (9)
O3—C17—O2	106.3 (5)	N3—C26—C27	123.3 (8)
O3—C17—C18	111.6 (6)	C23—C27—C26	118.4 (9)
O3—C17—C22	108.0 (6)	C29—C28—N2	124.3 (8)
C18—C17—C22	111.7 (6)	C29—C28—C32	120.1 (7)
C19—C18—C17	110.3 (6)	C32—C28—N2	115.6 (7)
C20—C19—C18	112.0 (7)	C28—C29—C30	117.3 (8)
C19—C20—C21	111.9 (7)	N4—C30—C29	124.7 (9)
C20—C21—C22	110.9 (8)	N4—C31—C32	123.1 (8)
C17—C22—C21	110.5 (7)	C28—C32—C31	118.1 (8)
C36—O4—C39	119.8 (6)	N6—N5—C55	112.3 (7)
C43—O5—C42	116.0 (6)	N5—N6—C60	113.2 (7)
C41—O8—C49	109.7 (6)	C57—N7—C58	118.3 (7)
C40—O9—C49	106.5 (5)	C63—N8—C62	118.5 (7)
C34—C33—I3	121.1 (6)	C56—C55—N5	124.7 (7)
C34—C33—C38	117.4 (7)	C56—C55—C59	120.1 (8)
C38—C33—I3	121.4 (5)	C59—C55—N5	115.1 (8)
F9—C34—C33	120.8 (8)	C55—C56—C57	117.6 (7)
F9—C34—C35	116.9 (8)	N7—C57—C56	123.6 (8)
C33—C34—C35	122.3 (8)	N7—C58—C59	122.7 (9)
F10—C35—C34	120.2 (8)	C55—C59—C58	117.6 (9)
F10—C35—C36	118.9 (7)	C61—C60—N6	116.0 (7)
C34—C35—C36	120.8 (7)	C61—C60—C64	119.8 (7)
O4—C36—C35	116.0 (6)	C64—C60—N6	124.3 (7)
O4—C36—C37	126.4 (7)	C60—C61—C62	116.8 (8)
C37—C36—C35	117.4 (7)	N8—C62—C61	123.4 (8)
F11—C37—C36	120.0 (7)	N8—C63—C64	123.0 (8)
F11—C37—C38	119.4 (7)	C63—C64—C60	118.5 (8)
I1—C1—C2—F1	0.3 (10)	O4—C39—C40—C41	54.0 (8)
I1—C1—C2—C3	-179.3 (5)	O5—C43—C44—F13	-1.5 (12)
I1—C1—C6—F4	-0.8 (10)	O5—C43—C44—C45	179.0 (7)
I1—C1—C6—C5	-178.2 (6)	O5—C43—C48—F16	2.2 (11)
I2—C14—C15—F7	4.1 (10)	O5—C43—C48—C47	-178.9 (7)
I2—C14—C15—C16	-174.3 (6)	O8—C41—C42—O5	-65.6 (8)
F6—C13—C14—I2	-5.5 (10)	O8—C49—C50—C51	-66.3 (9)

F6—C13—C14—C15	179.3 (7)	O8—C49—C54—C53	66.0 (9)
F5—C12—C13—F6	1.1 (10)	O9—C40—C41—O8	26.7 (7)
F5—C12—C13—C14	-179.0 (7)	O9—C40—C41—C42	144.5 (6)
F7—C15—C16—F8	0.8 (12)	O9—C49—C50—C51	179.1 (7)
F7—C15—C16—C11	-179.0 (7)	O9—C49—C54—C53	-177.9 (7)
F1—C2—C3—F2	-1.4 (10)	C33—C34—C35—F10	-179.7 (7)
F1—C2—C3—C4	178.4 (6)	C33—C34—C35—C36	-0.3 (12)
F2—C3—C4—O7	1.4 (10)	C34—C33—C38—F12	177.8 (7)
F2—C3—C4—C5	-177.6 (6)	C34—C33—C38—C37	1.3 (11)
F3—C5—C6—F4	-2.4 (11)	C34—C35—C36—O4	-174.2 (7)
F3—C5—C6—C1	175.0 (7)	C34—C35—C36—C37	0.2 (11)
O1—C11—C12—F5	3.8 (10)	C35—C36—C37—F11	-176.8 (7)
O1—C11—C12—C13	-175.1 (6)	C35—C36—C37—C38	0.7 (11)
O1—C11—C16—F8	-4.2 (10)	C36—O4—C39—C40	126.2 (7)
O1—C11—C16—C15	175.6 (7)	C36—C37—C38—F12	-178.2 (7)
O2—C9—C10—O1	-171.6 (5)	C36—C37—C38—C33	-1.5 (11)
O2—C17—C18—C19	177.3 (6)	C38—C33—C34—F9	179.5 (7)
O2—C17—C22—C21	-178.0 (7)	C38—C33—C34—C35	-0.4 (12)
O3—C8—C9—O2	33.1 (6)	C39—O4—C36—C35	-143.2 (7)
O3—C8—C9—C10	150.6 (6)	C39—O4—C36—C37	43.0 (11)
O3—C17—C18—C19	-65.3 (8)	C39—C40—C41—O8	141.9 (6)
O3—C17—C22—C21	66.5 (8)	C39—C40—C41—C42	-100.3 (7)
O7—C4—C5—F3	2.7 (10)	C40—O9—C49—O8	30.4 (7)
O7—C4—C5—C6	-179.0 (7)	C40—O9—C49—C50	147.3 (6)
O7—C7—C8—O3	-73.7 (7)	C40—O9—C49—C54	-88.4 (7)
O7—C7—C8—C9	174.1 (5)	C40—C41—C42—O5	179.9 (6)
C1—C2—C3—F2	178.2 (7)	C41—O8—C49—O9	-12.2 (8)
C1—C2—C3—C4	-2.0 (11)	C41—O8—C49—C50	-129.0 (7)
C2—C1—C6—F4	-178.9 (7)	C41—O8—C49—C54	107.4 (7)
C2—C1—C6—C5	3.8 (11)	C42—O5—C43—C44	58.9 (10)
C2—C3—C4—O7	-178.4 (6)	C42—O5—C43—C48	-126.5 (7)
C2—C3—C4—C5	2.5 (10)	C43—O5—C42—C41	69.7 (9)
C3—C4—C5—F3	-178.2 (6)	C43—C44—C45—F14	179.1 (7)
C3—C4—C5—C6	0.1 (11)	C43—C44—C45—C46	-1.4 (12)
C4—O7—C7—C8	64.9 (7)	C44—C43—C48—F16	177.2 (7)
C4—C5—C6—F4	179.4 (7)	C44—C43—C48—C47	-3.9 (11)
C4—C5—C6—C1	-3.3 (12)	C44—C45—C46—I4	178.0 (6)
C6—C1—C2—F1	178.4 (6)	C44—C45—C46—C47	-1.8 (11)
C6—C1—C2—C3	-1.2 (11)	C45—C46—C47—F15	-178.1 (7)
C7—O7—C4—C3	72.1 (8)	C45—C46—C47—C48	2.2 (11)

C7—O7—C4—C5	-108.9 (7)	C46—C47—C48—F16	179.6 (7)
C7—C8—C9—O2	149.1 (6)	C46—C47—C48—C43	0.7 (12)
C7—C8—C9—C10	-93.3 (7)	C48—C43—C44—F13	-176.4 (7)
C8—O3—C17—O2	26.7 (7)	C48—C43—C44—C45	4.2 (11)
C8—O3—C17—C18	-92.5 (7)	C49—O8—C41—C40	-8.8 (8)
C8—O3—C17—C22	144.5 (6)	C49—O8—C41—C42	-128.9 (7)
C8—C9—C10—O1	73.7 (7)	C49—O9—C40—C39	-155.5 (6)
C9—O2—C17—O3	-3.9 (7)	C49—O9—C40—C41	-35.0 (6)
C9—O2—C17—C18	116.7 (6)	C49—C50—C51—C52	-56.0 (10)
C9—O2—C17—C22	-120.4 (7)	C50—C49—C54—C53	-55.6 (10)
C10—O1—C11—C12	-73.4 (9)	C50—C51—C52—C53	57.0 (11)
C10—O1—C11—C16	111.0 (8)	C51—C52—C53—C54	-56.5 (11)
C11—O1—C10—C9	-169.4 (6)	C52—C53—C54—C49	54.6 (11)
C11—C12—C13—F6	180.0 (6)	C54—C49—C50—C51	55.7 (9)
C11—C12—C13—C14	-0.1 (11)	N1—N2—C28—C29	-2.6 (11)
C12—C11—C16—F8	180.0 (6)	N1—N2—C28—C32	178.3 (7)
C12—C11—C16—C15	-0.2 (11)	N1—C23—C24—C25	-179.2 (8)
C12—C13—C14—I2	174.5 (5)	N1—C23—C27—C26	179.8 (8)
C12—C13—C14—C15	-0.7 (11)	N2—N1—C23—C24	-5.6 (12)
C13—C14—C15—F7	179.4 (7)	N2—N1—C23—C27	175.5 (7)
C13—C14—C15—C16	1.0 (12)	N2—C28—C29—C30	-179.6 (8)
C14—C15—C16—F8	179.3 (7)	N2—C28—C32—C31	178.6 (8)
C14—C15—C16—C11	-0.5 (12)	N3—C26—C27—C23	0.3 (14)
C16—C11—C12—F5	179.4 (7)	N4—C31—C32—C28	0.8 (14)
C16—C11—C12—C13	0.5 (10)	C23—N1—N2—C28	177.9 (7)
C17—O2—C9—C8	-18.1 (7)	C23—C24—C25—N3	-1.3 (13)
C17—O2—C9—C10	-140.4 (6)	C24—C23—C27—C26	0.9 (13)
C17—O3—C8—C7	-158.2 (6)	C25—N3—C26—C27	-1.8 (13)
C17—O3—C8—C9	-36.7 (7)	C26—N3—C25—C24	2.4 (13)
C17—C18—C19—C20	-54.6 (10)	C27—C23—C24—C25	-0.3 (12)
C18—C17—C22—C21	-56.5 (9)	C28—C29—C30—N4	1.6 (14)
C18—C19—C20—C21	55.1 (11)	C29—C28—C32—C31	-0.5 (12)
C19—C20—C21—C22	-55.3 (11)	C30—N4—C31—C32	0.0 (14)
C20—C21—C22—C17	55.7 (10)	C31—N4—C30—C29	-1.3 (14)
C22—C17—C18—C19	55.6 (9)	C32—C28—C29—C30	-0.6 (12)
I3—C33—C34—F9	-4.1 (11)	N5—N6—C60—C61	-172.5 (8)
I3—C33—C34—C35	176.0 (6)	N5—N6—C60—C64	8.9 (12)
I3—C33—C38—F12	1.4 (10)	N5—C55—C56—C57	-180.0 (7)
I3—C33—C38—C37	-175.1 (5)	N5—C55—C59—C58	-179.5 (8)
I4—C46—C47—F15	2.1 (10)	N6—N5—C55—C56	0.5 (12)

I4—C46—C47—C48	-177.7 (6)	N6—N5—C55—C59	179.8 (8)
F11—C37—C38—F12	-0.6 (10)	N6—C60—C61—C62	179.4 (8)
F11—C37—C38—C33	176.0 (7)	N6—C60—C64—C63	179.2 (9)
F9—C34—C35—F10	0.4 (11)	N7—C58—C59—C55	-0.3 (15)
F9—C34—C35—C36	179.8 (7)	N8—C63—C64—C60	0.5 (15)
F10—C35—C36—O4	5.1 (10)	C55—N5—N6—C60	-179.8 (7)
F10—C35—C36—C37	179.5 (6)	C55—C56—C57—N7	-1.0 (12)
F15—C47—C48—F16	-0.1 (11)	C56—C55—C59—C58	-0.2 (14)
F15—C47—C48—C43	-179.0 (7)	C57—N7—C58—C59	0.1 (15)
F13—C44—C45—F14	-0.3 (11)	C58—N7—C57—C56	0.5 (13)
F13—C44—C45—C46	179.1 (7)	C59—C55—C56—C57	0.8 (12)
F14—C45—C46—I4	-2.5 (10)	C60—C61—C62—N8	2.3 (14)
F14—C45—C46—C47	177.6 (7)	C61—C60—C64—C63	0.7 (14)
O4—C36—C37—F11	-3.0 (11)	C62—N8—C63—C64	-0.2 (14)
O4—C36—C37—C38	174.5 (7)	C63—N8—C62—C61	-1.2 (14)
O4—C39—C40—O9	166.8 (5)	C64—C60—C61—C62	-1.9 (13)

apy·(*S*,*S*)-2

Crystal data

$C_{20}H_{14}F_8I_2O_2 \cdot C_{10}H_8N_4$	$Z = 4$
$M_r = 876.31$	$F(000) = 1696$
Orthorhombic, $P2_12_12_1$	$D_x = 1.905$ Mg m^{-3}
$a = 5.2031$ (2) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 17.8566$ (4) Å	$\mu = 2.14$ mm^{-1}
$c = 32.8945$ (8) Å	$T = 120$ K
$V = 3056.22$ (15) Å^3	

Data collection

38579 measured reflections	$\theta_{max} = 29.3°$, $\theta_{min} = 2.2°$
8255 independent reflections	$h = -7 \rightarrow 7$
6979 reflections with $I > 2\sigma(I)$	$k = -23 \rightarrow 24$
$R_{int} = 0.047$	$l = -45 \rightarrow 45$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 1.1536P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.091$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.08$	$\Delta\rangle_{max} = 1.32$ e Å^{-3}
8255 reflections	$\Delta\rangle_{min} = -0.62$ e Å^{-3}
415 parameters	Absolute structure: Flack x determined using 2672 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
0 restraints	Absolute structure parameter: -0.018 (15)
Primary atom site location: dual	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²) for **apy·(*S*,*S*)-2**

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.74442 (7)	0.35257 (2)	0.59112 (2)	0.02985 (9)
I2	0.44621 (7)	0.35961 (2)	0.90528 (2)	0.03136 (10)
F1	1.1225 (6)	0.41981 (19)	0.65979 (10)	0.0313 (7)
F2	1.1110 (6)	0.54715 (19)	0.70231 (11)	0.0348 (8)
F3	0.3876 (7)	0.62687 (18)	0.62724 (10)	0.0336 (8)
F4	0.3875 (6)	0.49796 (19)	0.58678 (10)	0.0355 (8)
F5	0.0874 (6)	0.50364 (18)	0.90871 (11)	0.0354 (7)
F6	0.0922 (6)	0.63458 (19)	0.87007 (10)	0.0341 (7)
F7	0.8238 (7)	0.55785 (19)	0.79634 (11)	0.0352 (8)
F8	0.8247 (7)	0.42726 (19)	0.83636 (10)	0.0333 (8)
O1	0.7635 (8)	0.6566 (2)	0.68679 (12)	0.0316 (8)
O2	0.4839 (7)	0.6671 (2)	0.81345 (12)	0.0292 (9)
C1	0.7531 (11)	0.4549 (3)	0.62222 (15)	0.0246 (10)
C2	0.9368 (11)	0.4706 (3)	0.65149 (16)	0.0260 (11)
C3	0.9331 (11)	0.5359 (3)	0.67323 (16)	0.0258 (11)
C4	0.7493 (12)	0.5907 (3)	0.66653 (16)	0.0266 (11)
C5	0.5661 (11)	0.5759 (3)	0.63631 (16)	0.0253 (11)
C6	0.5700 (11)	0.5088 (3)	0.61533 (15)	0.0254 (11)
C7	0.5395 (11)	0.6720 (3)	0.71164 (17)	0.0288 (11)
H7A	0.501264	0.628360	0.729164	0.035*

H7B	0.388489	0.681364	0.694035	0.035*
C8	0.5924 (10)	0.7406 (3)	0.73787 (16)	0.0231 (10)
H8	0.739815	0.729203	0.756376	0.028*
C9	0.6622 (11)	0.8083 (3)	0.71187 (17)	0.0264 (11)
H9A	0.819614	0.796908	0.696119	0.032*
H9B	0.521641	0.818004	0.692290	0.032*
C10	0.7075 (11)	0.8782 (3)	0.73724 (18)	0.0267 (11)
H10A	0.743446	0.921139	0.719081	0.032*
H10B	0.859125	0.870599	0.754949	0.032*
C11	0.4722 (11)	0.8955 (3)	0.76343 (18)	0.0279 (11)
H11A	0.509611	0.938917	0.781206	0.034*
H11B	0.325561	0.908937	0.745689	0.034*
C12	0.4013 (11)	0.8287 (3)	0.78926 (16)	0.0249 (11)
H12A	0.243827	0.840364	0.804907	0.030*
H12B	0.541329	0.818836	0.808922	0.030*
C13	0.3550 (10)	0.7583 (3)	0.76383 (16)	0.0218 (10)
H13	0.211779	0.770034	0.744598	0.026*
C14	0.2663 (11)	0.6931 (3)	0.78982 (16)	0.0257 (10)
H14A	0.125939	0.709307	0.808131	0.031*
H14B	0.200941	0.652201	0.772324	0.031*
C15	0.4590 (12)	0.5999 (3)	0.83187 (16)	0.0259 (11)
C16	0.2737 (12)	0.5831 (3)	0.86067 (16)	0.0272 (11)
C17	0.2719 (11)	0.5151 (3)	0.88088 (16)	0.0275 (11)
C18	0.4552 (12)	0.4612 (3)	0.87389 (16)	0.0277 (11)
C19	0.6410 (11)	0.4774 (3)	0.84480 (17)	0.0268 (11)
C20	0.6423 (11)	0.5448 (3)	0.82453 (16)	0.0262 (11)
N1	0.4995 (12)	0.0074 (3)	0.49588 (18)	0.0462 (14)
N2	0.2918 (13)	-0.0169 (4)	0.50279 (18)	0.0480 (15)
N3	0.6654 (11)	0.2147 (3)	0.55168 (16)	0.0338 (11)
N4	0.1284 (11)	-0.2221 (3)	0.44536 (16)	0.0352 (12)
C21	0.8066 (13)	0.1846 (4)	0.5225 (2)	0.0373 (14)
H21	0.955282	0.210650	0.513489	0.045*
C22	0.7471 (16)	0.1172 (4)	0.50470 (19)	0.0408 (14)
H22	0.852106	0.097588	0.483614	0.049*
C23	0.5378 (16)	0.0791 (3)	0.5174 (2)	0.0389 (15)
C24	0.3874 (14)	0.1088 (4)	0.5481 (2)	0.0451 (17)
H24	0.238857	0.083215	0.557518	0.054*
C25	0.4595 (13)	0.1772 (4)	0.56484 (19)	0.0353 (13)
H25	0.359406	0.197876	0.586224	0.042*
C26	-0.0102 (13)	-0.1931 (4)	0.4749 (2)	0.0401 (15)

H26	-0.157038	-0.220120	0.483840	0.048*
C27	0.0449 (16)	-0.1264 (4)	0.4935 (2)	0.0427 (15)
H27	-0.061775	-0.107491	0.514585	0.051*
C28	0.2568 (16)	-0.0879 (3)	0.48098 (19)	0.0388 (15)
C29	0.4051 (13)	-0.1168 (4)	0.4504 (2)	0.0385 (15)
H29	0.553975	-0.090929	0.441289	0.046*
C30	0.3347 (14)	-0.1839 (4)	0.43312 (19)	0.0378 (14)
H30	0.436652	-0.203728	0.411718	0.045*

Atomic displacement parameters (Å²) for **apy·(*S*,*S*)-2**

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.03950 (19)	0.02616 (16)	0.02388 (16)	0.00002 (15)	-0.00200 (15)	-0.00365 (14)
I2	0.0430 (2)	0.02658 (16)	0.02449 (16)	-0.00440 (15)	0.00430 (15)	0.00207 (15)
F1	0.0279 (17)	0.0310 (16)	0.0349 (18)	0.0018 (14)	-0.0043 (14)	-0.0017 (14)
F2	0.0257 (17)	0.0368 (18)	0.042 (2)	-0.0038 (15)	-0.0086 (15)	-0.0085 (15)
F3	0.0381 (19)	0.0317 (17)	0.0310 (17)	0.0097 (15)	-0.0017 (14)	-0.0012 (13)
F4	0.0390 (19)	0.0395 (18)	0.0280 (18)	0.0038 (15)	-0.0098 (15)	-0.0039 (14)
F5	0.0349 (17)	0.0390 (17)	0.0322 (17)	-0.0054 (14)	0.0120 (16)	0.0014 (15)
F6	0.0349 (18)	0.0345 (18)	0.0328 (17)	0.0035 (16)	0.0066 (14)	0.0011 (14)
F7	0.0289 (18)	0.0393 (19)	0.0375 (19)	-0.0061 (15)	0.0099 (15)	0.0068 (15)
F8	0.0305 (18)	0.0345 (18)	0.0348 (19)	0.0012 (15)	0.0046 (14)	0.0007 (14)
O1	0.0279 (19)	0.0279 (19)	0.039 (2)	-0.0046 (18)	0.0072 (18)	-0.0096 (16)
O2	0.025 (2)	0.0298 (19)	0.033 (2)	-0.0073 (16)	-0.0066 (16)	0.0066 (16)
C1	0.027 (3)	0.022 (2)	0.024 (2)	-0.001 (2)	0.002 (2)	0.0008 (18)
C2	0.027 (3)	0.023 (2)	0.028 (3)	0.001 (2)	0.005 (2)	0.000 (2)
C3	0.024 (3)	0.029 (3)	0.025 (3)	-0.006 (2)	0.000 (2)	-0.001 (2)
C4	0.028 (3)	0.025 (2)	0.027 (3)	-0.002 (3)	0.005 (2)	-0.0039 (19)
C5	0.029 (3)	0.024 (2)	0.024 (2)	0.002 (2)	0.002 (2)	0.0030 (19)
C6	0.026 (3)	0.032 (3)	0.018 (2)	-0.003 (2)	-0.001 (2)	0.0005 (19)
C7	0.024 (3)	0.029 (3)	0.033 (3)	-0.007 (2)	0.004 (2)	-0.004 (2)
C8	0.022 (3)	0.025 (2)	0.023 (2)	0.000 (2)	0.000 (2)	-0.0013 (19)
C9	0.024 (3)	0.029 (3)	0.026 (3)	-0.001 (2)	0.004 (2)	0.000 (2)
C10	0.025 (3)	0.020 (2)	0.035 (3)	-0.001 (2)	-0.001 (2)	0.002 (2)
C11	0.021 (3)	0.026 (3)	0.037 (3)	0.001 (2)	-0.001 (2)	-0.003 (2)
C12	0.023 (3)	0.026 (2)	0.025 (3)	0.000 (2)	0.001 (2)	-0.004 (2)
C13	0.019 (2)	0.024 (2)	0.022 (2)	-0.001 (2)	-0.0007 (19)	0.001 (2)
C14	0.017 (2)	0.034 (3)	0.026 (3)	-0.003 (2)	-0.004 (2)	0.004 (2)
C15	0.027 (3)	0.025 (2)	0.026 (3)	-0.006 (2)	-0.006 (2)	0.002 (2)
C16	0.027 (3)	0.029 (3)	0.025 (3)	0.002 (2)	-0.003 (2)	-0.002 (2)
C17	0.027 (3)	0.031 (3)	0.024 (2)	-0.003 (2)	0.002 (2)	-0.001 (2)

C18	0.033 (3)	0.026 (2)	0.024 (3)	-0.009 (2)	0.001 (2)	0.001 (2)
C19	0.027 (3)	0.027 (3)	0.027 (3)	-0.003 (2)	-0.001 (2)	-0.002 (2)
C20	0.024 (3)	0.035 (3)	0.019 (2)	-0.009 (2)	0.002 (2)	0.003 (2)
N1	0.038 (3)	0.053 (4)	0.048 (4)	0.005 (3)	0.004 (3)	0.008 (3)
N2	0.045 (4)	0.056 (4)	0.043 (3)	0.009 (3)	0.005 (3)	0.005 (3)
N3	0.040 (3)	0.027 (2)	0.035 (3)	0.001 (2)	-0.007 (2)	-0.001 (2)
N4	0.043 (3)	0.027 (2)	0.035 (3)	-0.005 (2)	-0.010 (2)	-0.002 (2)
C21	0.031 (3)	0.039 (3)	0.041 (4)	-0.001 (3)	0.001 (3)	0.002 (3)
C22	0.048 (4)	0.041 (3)	0.034 (3)	0.006 (3)	0.001 (3)	-0.002 (3)
C23	0.049 (4)	0.029 (3)	0.039 (3)	-0.003 (3)	-0.017 (3)	0.002 (2)
C24	0.039 (4)	0.042 (4)	0.055 (4)	-0.008 (3)	-0.012 (3)	0.014 (3)
C25	0.035 (3)	0.041 (3)	0.030 (3)	0.005 (3)	0.001 (3)	0.001 (2)
C26	0.034 (4)	0.043 (3)	0.043 (4)	0.001 (3)	-0.005 (3)	0.007 (3)
C27	0.049 (4)	0.044 (4)	0.035 (3)	0.011 (3)	0.002 (3)	-0.001 (3)
C28	0.049 (4)	0.032 (3)	0.035 (3)	0.009 (3)	-0.017 (3)	0.001 (2)
C29	0.035 (3)	0.037 (3)	0.044 (4)	-0.009 (3)	-0.005 (3)	0.008 (3)
C30	0.046 (4)	0.037 (3)	0.029 (3)	0.005 (3)	0.001 (3)	-0.002 (3)

Geometric parameters (Å, º) for **apy·(*S*,*S*)-2**

I1—C1	2.094 (5)	C11—C12	1.511 (8)
I2—C18	2.088 (5)	C12—C13	1.529 (7)
F1—C2	1.352 (6)	C13—C14	1.516 (7)
F2—C3	1.346 (6)	C15—C16	1.385 (8)
F3—C5	1.335 (6)	C15—C20	1.392 (8)
F4—C6	1.349 (6)	C16—C17	1.384 (8)
F5—C17	1.342 (6)	C17—C18	1.374 (8)
F6—C16	1.354 (6)	C18—C19	1.390 (8)
F7—C20	1.344 (6)	C19—C20	1.375 (8)
F8—C19	1.339 (7)	N1—N2	1.187 (8)
O1—C4	1.354 (6)	N1—C23	1.477 (9)
O1—C7	1.450 (7)	N2—C28	1.468 (9)
O2—C14	1.450 (6)	N3—C21	1.323 (8)
O2—C15	1.350 (6)	N3—C25	1.335 (9)
C1—C2	1.385 (8)	N4—C26	1.316 (9)
C1—C6	1.373 (8)	N4—C30	1.334 (9)
C2—C3	1.368 (7)	C21—C22	1.373 (9)
C3—C4	1.387 (8)	C22—C23	1.351 (11)
C4—C5	1.403 (8)	C23—C24	1.383 (10)
C5—C6	1.382 (7)	C24—C25	1.391 (9)
C7—C8	1.523 (7)	C26—C27	1.370 (10)

C8—C9	1.525 (7)	C27—C28	1.364 (12)
C8—C13	1.535 (7)	C28—C29	1.368 (10)
C9—C10	1.520 (7)	C29—C30	1.377 (9)
C10—C11	1.529 (8)		
C4—O1—C7	113.5 (4)	C16—C15—C20	116.2 (5)
C15—O2—C14	116.8 (4)	F6—C16—C15	119.6 (5)
C2—C1—I1	122.0 (4)	F6—C16—C17	118.8 (5)
C6—C1—I1	121.1 (4)	C17—C16—C15	121.6 (5)
C6—C1—C2	116.9 (5)	F5—C17—C16	117.8 (5)
F1—C2—C1	119.9 (5)	F5—C17—C18	120.3 (5)
F1—C2—C3	118.4 (5)	C18—C17—C16	121.9 (5)
C3—C2—C1	121.7 (5)	C17—C18—I2	120.7 (4)
F2—C3—C2	119.3 (5)	C17—C18—C19	116.9 (5)
F2—C3—C4	118.8 (5)	C19—C18—I2	122.4 (4)
C2—C3—C4	121.9 (5)	F8—C19—C18	120.0 (5)
O1—C4—C3	119.9 (5)	F8—C19—C20	118.7 (5)
O1—C4—C5	123.4 (5)	C20—C19—C18	121.3 (5)
C3—C4—C5	116.6 (5)	F7—C20—C15	118.6 (5)
F3—C5—C4	120.1 (5)	F7—C20—C19	119.3 (5)
F3—C5—C6	119.3 (5)	C19—C20—C15	122.1 (5)
C6—C5—C4	120.5 (5)	N2—N1—C23	110.4 (7)
F4—C6—C1	120.2 (5)	N1—N2—C28	109.6 (7)
F4—C6—C5	117.5 (5)	C21—N3—C25	118.5 (6)
C1—C6—C5	122.3 (5)	C26—N4—C30	117.6 (6)
O1—C7—C8	109.1 (4)	N3—C21—C22	122.7 (7)
C7—C8—C9	111.3 (4)	C23—C22—C21	119.4 (7)
C7—C8—C13	109.6 (4)	C22—C23—N1	113.3 (7)
C9—C8—C13	109.9 (4)	C22—C23—C24	119.3 (6)
C10—C9—C8	112.3 (4)	C24—C23—N1	127.3 (7)
C9—C10—C11	110.6 (5)	C23—C24—C25	118.2 (7)
C12—C11—C10	110.6 (4)	N3—C25—C24	121.9 (6)
C11—C12—C13	112.4 (4)	N4—C26—C27	123.8 (7)
C12—C13—C8	110.3 (4)	C28—C27—C26	118.2 (7)
C14—C13—C8	113.6 (4)	C27—C28—N2	112.8 (6)
C14—C13—C12	111.8 (4)	C27—C28—C29	119.2 (6)
O2—C14—C13	108.1 (4)	C29—C28—N2	127.9 (7)
O2—C15—C16	124.5 (5)	C28—C29—C30	118.8 (6)
O2—C15—C20	119.0 (5)	N4—C30—C29	122.3 (6)

I1—C1—C2—F1	2.0 (7)	C8—C13—C14—O2	53.3 (6)
I1—C1—C2—C3	-176.4 (4)	C9—C8—C13—C12	-55.4 (6)
I1—C1—C6—F4	-3.2 (7)	C9—C8—C13—C14	178.2 (4)
I1—C1—C6—C5	177.9 (4)	C9—C10—C11—C12	55.0 (7)
I2—C18—C19—F8	-0.8 (7)	C10—C11—C12—C13	-56.1 (6)
I2—C18—C19—C20	179.9 (4)	C11—C12—C13—C8	56.5 (6)
F1—C2—C3—F2	-0.8 (8)	C11—C12—C13—C14	-176.1 (5)
F1—C2—C3—C4	179.9 (5)	C12—C13—C14—O2	-72.3 (6)
F2—C3—C4—O1	5.3 (8)	C13—C8—C9—C10	56.5 (6)
F2—C3—C4—C5	-179.1 (5)	C14—O2—C15—C16	-60.5 (7)
F3—C5—C6—F4	-1.0 (8)	C14—O2—C15—C20	125.2 (6)
F3—C5—C6—C1	177.9 (5)	C15—O2—C14—C13	-166.3 (4)
F5—C17—C18—I2	1.4 (7)	C15—C16—C17—F5	179.2 (5)
F5—C17—C18—C19	-179.6 (5)	C15—C16—C17—C18	0.9 (9)
F6—C16—C17—F5	0.6 (8)	C16—C15—C20—F7	178.9 (5)
F6—C16—C17—C18	-177.8 (5)	C16—C15—C20—C19	-0.3 (8)
F8—C19—C20—F7	1.3 (8)	C16—C17—C18—I2	179.7 (4)
F8—C19—C20—C15	-179.5 (5)	C16—C17—C18—C19	-1.3 (8)
O1—C4—C5—F3	-2.6 (9)	C17—C18—C19—F8	-179.7 (5)
O1—C4—C5—C6	176.7 (5)	C17—C18—C19—C20	0.9 (8)
O1—C7—C8—C9	57.3 (6)	C18—C19—C20—F7	-179.3 (5)
O1—C7—C8—C13	179.1 (4)	C18—C19—C20—C15	-0.1 (9)
O2—C15—C16—F6	4.1 (9)	C20—C15—C16—F6	178.6 (5)
O2—C15—C16—C17	-174.5 (5)	C20—C15—C16—C17	0.0 (8)
O2—C15—C20—F7	-6.3 (8)	N1—N2—C28—C27	167.9 (6)
O2—C15—C20—C19	174.5 (5)	N1—N2—C28—C29	-13.5 (10)
C1—C2—C3—F2	177.7 (5)	N1—C23—C24—C25	179.1 (6)
C1—C2—C3—C4	-1.6 (9)	N2—N1—C23—C22	-167.4 (7)
C2—C1—C6—F4	178.9 (5)	N2—N1—C23—C24	13.1 (10)
C2—C1—C6—C5	0.0 (8)	N2—C28—C29—C30	-178.3 (6)
C2—C3—C4—O1	-175.4 (5)	N3—C21—C22—C23	-0.6 (10)
C2—C3—C4—C5	0.1 (8)	N4—C26—C27—C28	-0.6 (10)
C3—C4—C5—F3	-178.0 (5)	C21—N3—C25—C24	-1.5 (9)
C3—C4—C5—C6	1.3 (8)	C21—C22—C23—N1	-179.4 (6)
C4—O1—C7—C8	171.3 (5)	C21—C22—C23—C24	0.1 (10)
C4—C5—C6—F4	179.7 (5)	C22—C23—C24—C25	-0.3 (10)
C4—C5—C6—C1	-1.4 (9)	C23—N1—N2—C28	179.7 (5)
C6—C1—C2—F1	179.9 (5)	C23—C24—C25—N3	1.0 (9)
C6—C1—C2—C3	1.5 (8)	C25—N3—C21—C22	1.3 (10)
C7—O1—C4—C3	-120.2 (6)	C26—N4—C30—C29	0.4 (9)

C7—O1—C4—C5	64.5 (7)	C26—C27—C28—N2	179.0 (6)
C7—C8—C9—C10	178.1 (5)	C26—C27—C28—C29	0.3 (10)
C7—C8—C13—C12	-178.0 (4)	C27—C28—C29—C30	0.2 (10)
C7—C8—C13—C14	55.6 (6)	C28—C29—C30—N4	-0.6 (10)
C8—C9—C10—C11	-56.3 (6)	C30—N4—C26—C27	0.2 (10)

apy·(*R*)-3

Crystal data

$C_{32}H_{12}F_8I_2O_2 \cdot C_{10}H_8N_4$	$Z = 4$
$M_r = 1018.42$	$F(000) = 1976$
Orthorhombic, $P2_12_12_1$	$D_x = 1.811$ Mg m^{-3}
$a = 11.5438$ (5) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 14.8242$ (7) Å	$\mu = 1.77$ mm^{-1}
$c = 21.8290$ (17) Å	$T = 120$ K
$V = 3735.5$ (4) Å^3	

Data collection

54653 measured reflections	$\theta_{max} = 29.3°$, $\theta_{min} = 2.2°$
10101 independent reflections	$h = -15 \rightarrow 15$
8360 reflections with $I > 2\sigma(I)$	$k = -20 \rightarrow 20$
$R_{int} = 0.052$	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2 + 2.0256P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.123$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.09$	$\Delta\rangle_{max} = 0.96$ e Å^{-3}
10101 reflections	$\Delta\rangle_{min} = -1.24$ e Å^{-3}
523 parameters	Absolute structure: Flack x determined using 3261 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
0 restraints	Absolute structure parameter: -0.030 (14)
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²) for **apy·(*R*)-3**

	x	*y*	*Z*	U_{iso}*/U_{eq}
I1	-0.19490 (4)	0.28021 (3)	0.14097 (2)	0.04082 (12)
I2	0.78041 (4)	-0.15959 (4)	0.06938 (2)	0.05196 (15)
F1	-0.0738 (4)	0.1657 (3)	0.03667 (17)	0.0455 (10)
F2	0.0663 (4)	0.0218 (3)	0.03886 (19)	0.0457 (10)
F3	-0.0933 (4)	0.1580 (3)	0.25316 (18)	0.0448 (9)
F4	0.0510 (4)	0.0163 (3)	0.25436 (19)	0.0440 (10)
F5	0.7272 (4)	0.0529 (3)	0.0546 (2)	0.0544 (11)
F6	0.5302 (4)	0.1447 (3)	0.0389 (2)	0.0482 (10)
F7	0.5147 (5)	-0.2190 (3)	0.0627 (2)	0.0567 (11)
F8	0.3188 (4)	-0.1262 (3)	0.0436 (2)	0.0517 (11)
O1	0.1370 (4)	-0.0549 (3)	0.1455 (3)	0.0408 (10)
O2	0.3185 (4)	0.0554 (4)	0.0313 (2)	0.0434 (12)
C1	-0.0857 (5)	0.1666 (5)	0.1447 (3)	0.0378 (13)
C2	-0.0442 (6)	0.1297 (5)	0.0911 (3)	0.0392 (15)
C3	0.0278 (6)	0.0564 (5)	0.0920 (3)	0.0367 (14)
C4	0.0645 (6)	0.0184 (5)	0.1468 (3)	0.0373 (13)
C5	0.0203 (6)	0.0537 (5)	0.2011 (3)	0.0358 (14)
C6	-0.0540 (6)	0.1274 (5)	0.1992 (3)	0.0376 (14)
C7	0.6262 (6)	-0.0866 (6)	0.0596 (3)	0.0437 (16)
C8	0.5227 (7)	-0.1285 (5)	0.0562 (3)	0.0438 (16)
C9	0.4201 (6)	-0.0818 (5)	0.0473 (3)	0.0416 (16)
C10	0.4207 (6)	0.0111 (5)	0.0425 (3)	0.0390 (15)
C11	0.5272 (7)	0.0555 (5)	0.0449 (3)	0.0418 (16)
C12	0.6278 (6)	0.0074 (5)	0.0530 (3)	0.0418 (16)
C13	0.2495 (6)	-0.0491 (5)	0.1691 (3)	0.0342 (13)
C14	0.2870 (7)	-0.1322 (4)	0.1949 (3)	0.0383 (14)
H14	0.235363	-0.181978	0.197211	0.046*
C15	0.3970 (7)	-0.1401 (5)	0.2161 (3)	0.0429 (16)
H15	0.423042	-0.196299	0.231835	0.051*
C16	0.4730 (6)	-0.0659 (5)	0.2151 (3)	0.0376 (14)
C17	0.5882 (7)	-0.0723 (6)	0.2373 (3)	0.0442 (17)
H17	0.615894	-0.128619	0.252060	0.053*

C18	0.6607 (7)	0.0015 (6)	0.2378 (3)	0.0480 (18)
H18	0.737357	-0.003817	0.253184	0.058*
C19	0.6208 (6)	0.0843 (6)	0.2154 (3)	0.0432 (16)
H19	0.670171	0.135558	0.216404	0.052*
C20	0.5108 (6)	0.0919 (5)	0.1922 (3)	0.0381 (14)
H20	0.485948	0.148627	0.176690	0.046*
C21	0.4340 (6)	0.0189 (5)	0.1905 (3)	0.0358 (14)
C22	0.3196 (6)	0.0253 (5)	0.1649 (3)	0.0335 (13)
C23	0.2832 (5)	0.1105 (4)	0.1342 (3)	0.0342 (12)
C24	0.2846 (6)	0.1225 (5)	0.0721 (3)	0.0373 (13)
C25	0.2419 (6)	0.2014 (5)	0.0440 (3)	0.0443 (17)
H25	0.244852	0.207310	0.000720	0.053*
C26	0.1961 (6)	0.2697 (5)	0.0788 (3)	0.0458 (16)
H26	0.166395	0.322196	0.059411	0.055*
C27	0.1929 (5)	0.2621 (5)	0.1431 (3)	0.0408 (14)
C28	0.1407 (7)	0.3280 (5)	0.1807 (4)	0.0486 (18)
H28	0.109174	0.380546	0.162202	0.058*
C29	0.1340 (7)	0.3189 (5)	0.2416 (4)	0.0506 (19)
H29	0.096231	0.363775	0.265350	0.061*
C30	0.1826 (7)	0.2429 (5)	0.2705 (3)	0.0448 (16)
H30	0.179815	0.237431	0.313836	0.054*
C31	0.2340 (6)	0.1770 (5)	0.2361 (3)	0.0391 (14)
H31	0.267036	0.126046	0.255903	0.047*
C32	0.2387 (6)	0.1834 (4)	0.1713 (3)	0.0343 (13)
N1	-0.6523 (6)	0.5829 (4)	0.1097 (3)	0.0429 (14)
N2	-0.7489 (5)	0.5461 (4)	0.1035 (3)	0.0405 (13)
N3	-0.3626 (6)	0.4147 (5)	0.1250 (3)	0.0480 (15)
N4	-1.0294 (6)	0.7241 (6)	0.0853 (3)	0.0539 (17)
C33	-0.5576 (7)	0.5219 (5)	0.1126 (3)	0.0409 (15)
C34	-0.5640 (7)	0.4313 (5)	0.0965 (3)	0.0443 (17)
H34	-0.633818	0.405034	0.081959	0.053*
C35	-0.4644 (7)	0.3817 (6)	0.1027 (4)	0.0504 (19)
H35	-0.466597	0.320129	0.090723	0.060*
C36	-0.3623 (7)	0.5006 (6)	0.1411 (4)	0.0511 (18)
H36	-0.292697	0.524276	0.157811	0.061*
C37	-0.4543 (7)	0.5580 (6)	0.1355 (4)	0.0502 (18)
H37	-0.448376	0.619696	0.146795	0.060*
C38	-0.8419 (7)	0.6090 (5)	0.0982 (3)	0.0382 (14)
C39	-0.8253 (7)	0.7013 (5)	0.0876 (3)	0.0443 (17)
H39	-0.749708	0.726441	0.085224	0.053*

C40	-0.9222 (8)	0.7543 (6)	0.0809 (4)	0.052 (2)
H40	-0.911311	0.816567	0.072426	0.063*
C41	-1.0434 (8)	0.6358 (6)	0.0962 (4)	0.0496 (19)
H41	-1.120176	0.613440	0.100040	0.059*
C42	-0.9532 (7)	0.5756 (6)	0.1020 (3)	0.0454 (18)
H42	-0.967230	0.513186	0.108427	0.054*

Atomic displacement parameters (Å^2) for **apy·(*R*)-3**

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0352 (2)	0.0518 (2)	0.0355 (2)	0.0076 (2)	-0.00003 (17)	-0.00177 (19)
I2	0.0482 (3)	0.0712 (3)	0.0364 (2)	0.0215 (2)	-0.00374 (19)	-0.0025 (2)
F1	0.044 (2)	0.063 (3)	0.0299 (18)	0.007 (2)	-0.0040 (16)	0.0020 (19)
F2	0.043 (2)	0.059 (3)	0.035 (2)	0.010 (2)	0.0017 (18)	-0.0055 (19)
F3	0.040 (2)	0.061 (3)	0.0335 (19)	0.001 (2)	0.0034 (16)	-0.0018 (19)
F4	0.043 (2)	0.053 (2)	0.036 (2)	0.002 (2)	-0.0020 (17)	0.0062 (18)
F5	0.032 (2)	0.068 (3)	0.063 (3)	0.001 (2)	0.004 (2)	-0.003 (2)
F6	0.042 (2)	0.048 (2)	0.055 (3)	0.004 (2)	0.0050 (19)	0.010 (2)
F7	0.070 (3)	0.043 (2)	0.057 (3)	0.004 (2)	0.012 (2)	-0.006 (2)
F8	0.040 (2)	0.061 (3)	0.054 (2)	-0.005 (2)	0.007 (2)	-0.007 (2)
O1	0.035 (2)	0.040 (2)	0.048 (3)	0.0019 (19)	-0.001 (2)	-0.002 (2)
O2	0.032 (2)	0.065 (3)	0.033 (2)	0.008 (2)	-0.0032 (19)	-0.003 (2)
C1	0.031 (3)	0.040 (3)	0.042 (3)	0.003 (3)	-0.001 (3)	0.002 (3)
C2	0.029 (3)	0.056 (4)	0.033 (3)	0.002 (3)	-0.002 (2)	-0.003 (3)
C3	0.031 (3)	0.047 (4)	0.033 (3)	0.003 (3)	0.000 (2)	-0.005 (3)
C4	0.028 (3)	0.043 (3)	0.041 (3)	0.002 (3)	-0.003 (3)	0.000 (3)
C5	0.030 (3)	0.045 (4)	0.033 (3)	-0.002 (3)	0.002 (2)	0.007 (3)
C6	0.031 (3)	0.048 (4)	0.033 (3)	-0.003 (3)	0.004 (2)	-0.006 (3)
C7	0.039 (3)	0.062 (5)	0.030 (3)	0.012 (3)	-0.003 (3)	-0.001 (3)
C8	0.050 (4)	0.046 (4)	0.035 (3)	0.007 (3)	0.005 (3)	0.000 (3)
C9	0.034 (3)	0.056 (4)	0.035 (3)	0.000 (3)	0.002 (3)	-0.009 (3)
C10	0.030 (3)	0.057 (4)	0.030 (3)	0.007 (3)	0.003 (2)	0.001 (3)
C11	0.038 (4)	0.050 (4)	0.037 (3)	0.000 (3)	0.005 (3)	0.002 (3)
C12	0.033 (3)	0.054 (4)	0.038 (3)	0.001 (3)	0.002 (3)	0.002 (3)
C13	0.031 (3)	0.038 (3)	0.033 (3)	0.006 (3)	0.000 (2)	0.001 (2)
C14	0.044 (4)	0.035 (3)	0.036 (3)	0.003 (3)	0.000 (3)	0.001 (2)
C15	0.048 (4)	0.043 (4)	0.038 (3)	0.007 (3)	-0.002 (3)	0.006 (3)
C16	0.039 (3)	0.043 (4)	0.031 (3)	0.005 (3)	-0.001 (3)	0.000 (3)
C17	0.040 (4)	0.053 (4)	0.040 (4)	0.011 (3)	0.000 (3)	0.006 (3)

C18	0.038 (4)	0.069 (5)	0.037 (4)	0.011 (4)	-0.006 (3)	-0.005 (3)
C19	0.030 (3)	0.058 (4)	0.042 (4)	-0.003 (3)	-0.002 (3)	0.005 (3)
C20	0.037 (3)	0.043 (4)	0.035 (3)	0.006 (3)	0.002 (3)	0.006 (3)
C21	0.032 (3)	0.046 (4)	0.029 (3)	0.003 (3)	0.003 (2)	0.001 (3)
C22	0.033 (3)	0.039 (3)	0.029 (3)	0.004 (3)	0.001 (2)	0.003 (2)
C23	0.028 (3)	0.043 (3)	0.032 (3)	0.002 (2)	-0.003 (2)	0.003 (2)
C24	0.031 (3)	0.048 (4)	0.034 (3)	0.005 (3)	0.002 (3)	0.006 (3)
C25	0.036 (3)	0.058 (5)	0.039 (3)	0.007 (3)	0.003 (3)	0.012 (3)
C26	0.035 (3)	0.050 (4)	0.052 (4)	-0.001 (3)	-0.007 (3)	0.018 (3)
C27	0.025 (3)	0.044 (3)	0.053 (4)	0.002 (3)	-0.003 (3)	0.007 (3)
C28	0.042 (4)	0.035 (4)	0.069 (5)	-0.005 (3)	-0.005 (3)	0.006 (3)
C29	0.042 (4)	0.039 (4)	0.071 (5)	-0.001 (3)	0.003 (4)	-0.010 (4)
C30	0.045 (4)	0.046 (4)	0.043 (4)	-0.005 (3)	0.002 (3)	-0.004 (3)
C31	0.037 (3)	0.037 (3)	0.044 (3)	-0.003 (3)	0.005 (3)	-0.002 (3)
C32	0.026 (3)	0.040 (3)	0.037 (3)	0.000 (3)	0.000 (2)	0.007 (2)
N1	0.042 (3)	0.044 (3)	0.043 (3)	0.005 (3)	-0.002 (3)	0.002 (3)
N2	0.041 (3)	0.046 (3)	0.035 (3)	0.004 (3)	0.002 (2)	0.001 (2)
N3	0.041 (3)	0.062 (4)	0.041 (3)	0.008 (3)	0.002 (3)	0.000 (3)
N4	0.050 (4)	0.072 (5)	0.040 (3)	0.018 (4)	-0.006 (3)	-0.009 (3)
C33	0.042 (4)	0.047 (4)	0.034 (3)	0.006 (3)	-0.004 (3)	0.004 (3)
C34	0.041 (4)	0.052 (4)	0.039 (4)	0.004 (3)	0.001 (3)	-0.006 (3)
C35	0.046 (4)	0.060 (5)	0.045 (4)	0.013 (4)	-0.003 (3)	-0.007 (4)
C36	0.039 (4)	0.062 (5)	0.053 (4)	-0.001 (3)	-0.005 (4)	-0.004 (4)
C37	0.042 (4)	0.054 (4)	0.054 (5)	0.002 (3)	0.002 (4)	-0.004 (4)
C38	0.039 (3)	0.043 (4)	0.032 (3)	0.004 (3)	0.002 (3)	0.001 (3)
C39	0.048 (4)	0.044 (4)	0.041 (3)	0.006 (3)	-0.006 (3)	-0.010 (3)
C40	0.052 (4)	0.053 (4)	0.052 (5)	0.012 (4)	-0.008 (4)	-0.010 (3)
C41	0.045 (4)	0.063 (5)	0.040 (4)	0.006 (4)	0.001 (3)	0.003 (3)
C42	0.037 (4)	0.064 (5)	0.035 (3)	0.006 (4)	0.005 (3)	0.003 (3)

Geometric parameters (Å, º) for **apy·(*R*)-3**

I1—C1	2.106 (7)	C21—C22	1.436 (9)
I2—C7	2.094 (7)	C22—C23	1.491 (9)
F1—C2	1.348 (8)	C23—C24	1.367 (9)
F2—C3	1.344 (8)	C23—C32	1.445 (9)
F3—C6	1.342 (8)	C24—C25	1.409 (10)
F4—C5	1.335 (8)	C25—H25	0.9500
F5—C12	1.332 (9)	C25—C26	1.370 (11)
F6—C11	1.330 (9)	C26—H26	0.9500
F7—C8	1.352 (9)	C26—C27	1.409 (10)

F8—C9	1.344 (9)	C27—C28	1.411 (11)
O1—C4	1.371 (8)	C27—C32	1.422 (9)
O1—C13	1.400 (8)	C28—H28	0.9500
O2—C10	1.372 (8)	C28—C29	1.338 (12)
O2—C24	1.392 (8)	C29—H29	0.9500
C1—C2	1.378 (10)	C29—C30	1.408 (11)
C1—C6	1.372 (10)	C30—H30	0.9500
C2—C3	1.367 (10)	C30—C31	1.368 (10)
C3—C4	1.390 (10)	C31—H31	0.9500
C4—C5	1.393 (10)	C31—C32	1.419 (9)
C5—C6	1.389 (10)	N1—N2	1.248 (9)
C7—C8	1.349 (12)	N1—C33	1.420 (10)
C7—C12	1.401 (12)	N2—C38	1.427 (10)
C8—C9	1.386 (11)	N3—C35	1.362 (11)
C9—C10	1.382 (11)	N3—C36	1.321 (11)
C10—C11	1.395 (11)	N4—C40	1.319 (12)
C11—C12	1.373 (11)	N4—C41	1.340 (12)
C13—C14	1.423 (9)	C33—C34	1.390 (11)
C13—C22	1.371 (10)	C33—C37	1.400 (11)
C14—H14	0.9500	C34—H34	0.9500
C14—C15	1.357 (11)	C34—C35	1.371 (11)
C15—H15	0.9500	C35—H35	0.9500
C15—C16	1.407 (11)	C36—H36	0.9500
C16—C17	1.418 (10)	C36—C37	1.366 (11)
C16—C21	1.439 (10)	C37—H37	0.9500
C17—H17	0.9500	C38—C39	1.401 (10)
C17—C18	1.378 (12)	C38—C42	1.380 (11)
C18—H18	0.9500	C39—H39	0.9500
C18—C19	1.399 (11)	C39—C40	1.375 (11)
C19—H19	0.9500	C40—H40	0.9500
C19—C20	1.373 (10)	C41—H41	0.9500
C20—H20	0.9500	C41—C42	1.377 (12)
C20—C21	1.400 (10)	C42—H42	0.9500
C4—O1—C13	120.6 (5)	C13—C22—C23	123.0 (6)
C10—O2—C24	118.0 (5)	C21—C22—C23	119.4 (6)
C2—C1—I1	119.5 (5)	C24—C23—C22	123.6 (6)
C6—C1—I1	122.2 (5)	C24—C23—C32	117.6 (6)
C6—C1—C2	118.3 (6)	C32—C23—C22	118.8 (5)
F1—C2—C1	120.2 (6)	O2—C24—C25	114.4 (6)

F1—C2—C3	118.7 (6)	C23—C24—O2	123.0 (6)
C3—C2—C1	121.0 (6)	C23—C24—C25	122.3 (6)
F2—C3—C2	119.5 (6)	C24—C25—H25	119.8
F2—C3—C4	119.2 (6)	C26—C25—C24	120.5 (6)
C2—C3—C4	121.3 (6)	C26—C25—H25	119.8
O1—C4—C3	119.3 (6)	C25—C26—H26	119.9
O1—C4—C5	122.7 (6)	C25—C26—C27	120.2 (6)
C3—C4—C5	117.9 (6)	C27—C26—H26	119.9
F4—C5—C4	119.1 (6)	C26—C27—C28	122.5 (7)
F4—C5—C6	121.2 (6)	C26—C27—C32	119.2 (7)
C6—C5—C4	119.7 (6)	C28—C27—C32	118.3 (7)
F3—C6—C1	121.8 (6)	C27—C28—H28	118.9
F3—C6—C5	116.6 (6)	C29—C28—C27	122.2 (7)
C1—C6—C5	121.7 (6)	C29—C28—H28	118.9
C8—C7—I2	121.4 (6)	C28—C29—H29	119.9
C8—C7—C12	117.7 (7)	C28—C29—C30	120.2 (8)
C12—C7—I2	120.9 (6)	C30—C29—H29	119.9
F7—C8—C9	116.9 (8)	C29—C30—H30	120.1
C7—C8—F7	120.8 (7)	C31—C30—C29	119.9 (7)
C7—C8—C9	122.3 (7)	C31—C30—H30	120.1
F8—C9—C8	120.5 (7)	C30—C31—H31	119.5
F8—C9—C10	119.2 (7)	C30—C31—C32	121.1 (7)
C10—C9—C8	120.3 (7)	C32—C31—H31	119.5
O2—C10—C9	119.1 (7)	C27—C32—C23	120.2 (6)
O2—C10—C11	122.6 (7)	C31—C32—C23	121.5 (6)
C9—C10—C11	118.1 (7)	C31—C32—C27	118.2 (6)
F6—C11—C10	119.2 (7)	N2—N1—C33	114.4 (6)
F6—C11—C12	120.5 (7)	N1—N2—C38	113.2 (6)
C12—C11—C10	120.3 (7)	C36—N3—C35	116.3 (7)
F5—C12—C7	120.8 (7)	C40—N4—C41	117.2 (8)
F5—C12—C11	118.0 (7)	C34—C33—N1	124.3 (7)
C11—C12—C7	121.2 (7)	C34—C33—C37	120.3 (7)
O1—C13—C14	112.0 (6)	C37—C33—N1	115.4 (7)
C22—C13—O1	125.0 (6)	C33—C34—H34	121.7
C22—C13—C14	122.9 (6)	C35—C34—C33	116.7 (8)
C13—C14—H14	120.2	C35—C34—H34	121.7
C15—C14—C13	119.7 (7)	N3—C35—C34	124.5 (8)
C15—C14—H14	120.2	N3—C35—H35	117.8
C14—C15—H15	119.6	C34—C35—H35	117.8
C14—C15—C16	120.7 (7)	N3—C36—H36	117.5

C16—C15—H15	119.6	N3—C36—C37	125.1 (8)
C15—C16—C17	121.8 (7)	C37—C36—H36	117.5
C15—C16—C21	119.5 (6)	C33—C37—H37	121.4
C17—C16—C21	118.7 (7)	C36—C37—C33	117.1 (8)
C16—C17—H17	119.4	C36—C37—H37	121.4
C18—C17—C16	121.2 (7)	C39—C38—N2	123.3 (7)
C18—C17—H17	119.4	C42—C38—N2	117.5 (7)
C17—C18—H18	120.2	C42—C38—C39	119.2 (8)
C17—C18—C19	119.6 (7)	C38—C39—H39	121.2
C19—C18—H18	120.2	C40—C39—C38	117.7 (8)
C18—C19—H19	119.8	C40—C39—H39	121.2
C20—C19—C18	120.4 (7)	N4—C40—C39	124.2 (8)
C20—C19—H19	119.8	N4—C40—H40	117.9
C19—C20—H20	118.9	C39—C40—H40	117.9
C19—C20—C21	122.1 (7)	N4—C41—H41	118.0
C21—C20—H20	118.9	N4—C41—C42	123.9 (9)
C20—C21—C16	117.8 (6)	C42—C41—H41	118.0
C20—C21—C22	122.7 (6)	C38—C42—H42	121.1
C22—C21—C16	119.4 (6)	C41—C42—C38	117.8 (8)
C13—C22—C21	117.6 (6)	C41—C42—H42	121.1
I1—C1—C2—F1	-0.4 (9)	C15—C16—C17—C18	-178.0 (7)
I1—C1—C2—C3	179.3 (5)	C15—C16—C21—C20	177.9 (6)
I1—C1—C6—F3	1.8 (9)	C15—C16—C21—C22	-2.6 (10)
I1—C1—C6—C5	-178.5 (5)	C16—C17—C18—C19	-0.7 (12)
I2—C7—C8—F7	4.0 (10)	C16—C21—C22—C13	4.4 (9)
I2—C7—C8—C9	-177.3 (5)	C16—C21—C22—C23	-174.4 (6)
I2—C7—C12—F5	-2.1 (10)	C17—C16—C21—C20	-2.8 (10)
I2—C7—C12—C11	178.3 (5)	C17—C16—C21—C22	176.8 (6)
F1—C2—C3—F2	-0.6 (10)	C17—C18—C19—C20	-1.1 (12)
F1—C2—C3—C4	177.7 (6)	C18—C19—C20—C21	0.9 (11)
F2—C3—C4—O1	-1.6 (10)	C19—C20—C21—C16	1.1 (10)
F2—C3—C4—C5	-178.0 (6)	C19—C20—C21—C22	-178.5 (7)
F4—C5—C6—F3	-0.4 (10)	C20—C21—C22—C13	-176.1 (6)
F4—C5—C6—C1	179.7 (6)	C20—C21—C22—C23	5.1 (10)
F6—C11—C12—F5	0.1 (11)	C21—C16—C17—C18	2.7 (11)
F6—C11—C12—C7	179.7 (7)	C21—C22—C23—C24	98.8 (8)
F7—C8—C9—F8	-1.9 (10)	C21—C22—C23—C32	-84.2 (8)
F7—C8—C9—C10	177.7 (6)	C22—C13—C14—C15	-0.5 (10)
F8—C9—C10—O2	-3.0 (10)	C22—C23—C24—O2	1.2 (10)

F8—C9—C10—C11	-178.5 (6)	C22—C23—C24—C25	175.3 (6)
O1—C4—C5—F4	1.4 (10)	C22—C23—C32—C27	-174.0 (6)
O1—C4—C5—C6	-179.1 (6)	C22—C23—C32—C31	2.9 (9)
O1—C13—C14—C15	175.6 (6)	C23—C24—C25—C26	-0.4 (11)
O1—C13—C22—C21	-178.6 (6)	C24—O2—C10—C9	124.2 (7)
O1—C13—C22—C23	0.2 (10)	C24—O2—C10—C11	-60.4 (9)
O2—C10—C11—F6	3.1 (11)	C24—C23—C32—C27	3.2 (9)
O2—C10—C11—C12	-176.4 (7)	C24—C23—C32—C31	-179.9 (6)
O2—C24—C25—C26	174.3 (6)	C24—C25—C26—C27	1.0 (11)
C1—C2—C3—F2	179.7 (6)	C25—C26—C27—C28	-176.2 (7)
C1—C2—C3—C4	-2.0 (11)	C25—C26—C27—C32	0.6 (10)
C2—C1—C6—F3	-178.2 (7)	C26—C27—C28—C29	177.4 (8)
C2—C1—C6—C5	1.6 (10)	C26—C27—C32—C23	-2.6 (10)
C2—C3—C4—O1	-179.8 (6)	C26—C27—C32—C31	-179.6 (6)
C2—C3—C4—C5	3.7 (10)	C27—C28—C29—C30	1.7 (12)
C3—C4—C5—F4	177.6 (7)	C28—C27—C32—C23	174.2 (6)
C3—C4—C5—C6	-2.8 (10)	C28—C27—C32—C31	-2.7 (10)
C4—O1—C13—C14	147.5 (6)	C28—C29—C30—C31	-1.9 (12)
C4—O1—C13—C22	-36.4 (9)	C29—C30—C31—C32	-0.3 (11)
C4—C5—C6—F3	-180.0 (6)	C30—C31—C32—C23	-174.3 (7)
C4—C5—C6—C1	0.2 (10)	C30—C31—C32—C27	2.6 (10)
C6—C1—C2—F1	179.6 (6)	C32—C23—C24—O2	-175.9 (6)
C6—C1—C2—C3	-0.7 (10)	C32—C23—C24—C25	-1.7 (10)
C7—C8—C9—F8	179.4 (7)	C32—C27—C28—C29	0.6 (11)
C7—C8—C9—C10	-1.1 (11)	N1—N2—C38—C39	13.1 (10)
C8—C7—C12—F5	-178.6 (6)	N1—N2—C38—C42	-169.0 (6)
C8—C7—C12—C11	1.7 (11)	N1—C33—C34—C35	-179.3 (7)
C8—C9—C10—O2	177.5 (6)	N1—C33—C37—C36	177.8 (7)
C8—C9—C10—C11	2.0 (11)	N2—N1—C33—C34	14.0 (10)
C9—C10—C11—F6	178.4 (6)	N2—N1—C33—C37	-163.6 (7)
C9—C10—C11—C12	-1.1 (11)	N2—C38—C39—C40	177.3 (7)
C10—O2—C24—C23	-49.1 (9)	N2—C38—C42—C41	-179.2 (6)
C10—O2—C24—C25	136.3 (7)	N3—C36—C37—C33	1.9 (14)
C10—C11—C12—F5	179.6 (7)	N4—C41—C42—C38	2.0 (12)
C10—C11—C12—C7	-0.8 (11)	C33—N1—N2—C38	-177.6 (6)
C12—C7—C8—F7	-179.5 (6)	C33—C34—C35—N3	2.0 (13)
C12—C7—C8—C9	-0.8 (11)	C34—C33—C37—C36	0.1 (12)
C13—O1—C4—C3	115.7 (7)	C35—N3—C36—C37	-1.8 (13)
C13—O1—C4—C5	-68.1 (9)	C36—N3—C35—C34	-0.2 (13)
C13—C14—C15—C16	2.5 (11)	C37—C33—C34—C35	-1.8 (12)

C13—C22—C23—C24	-79.9 (9)	C38—C39—C40—N4	1.9 (12)
C13—C22—C23—C32	97.1 (8)	C39—C38—C42—C41	-1.1 (11)
C14—C13—C22—C21	-2.9 (9)	C40—N4—C41—C42	-0.8 (12)
C14—C13—C22—C23	175.8 (6)	C41—N4—C40—C39	-1.2 (12)
C14—C15—C16—C17	179.8 (7)	C42—C38—C39—C40	-0.6 (11)
C14—C15—C16—C21	-0.9 (10)		

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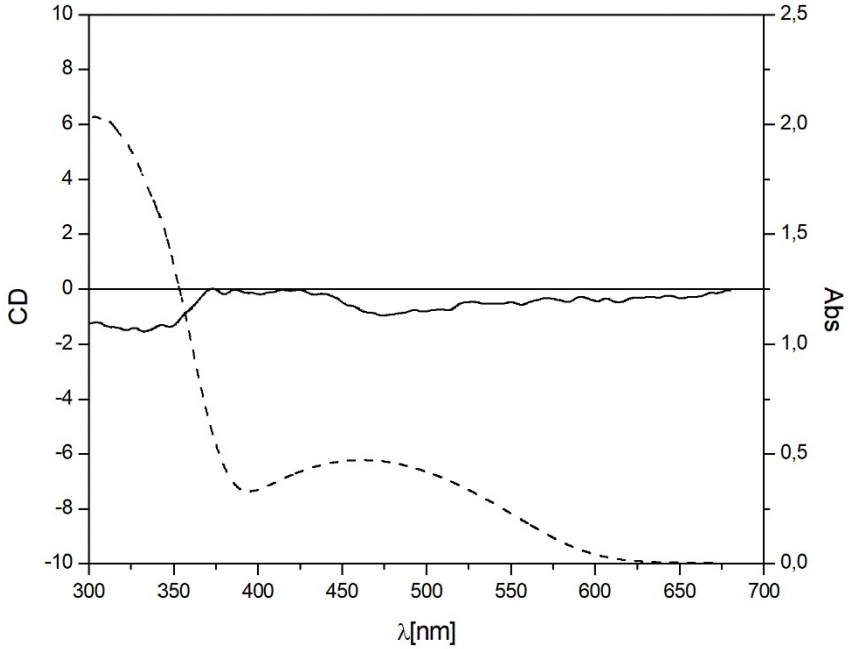

Figure S16. CD (solid line) and UV-vis (dashed line) spectra of **apy·(*S*,*S*)-1b** measured in KBr disk.

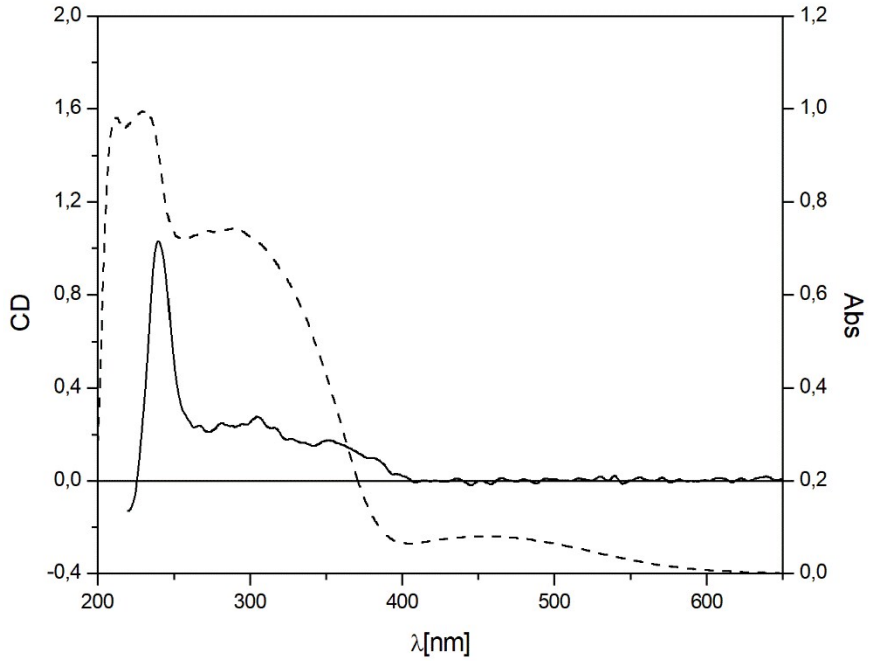

Figure S17. CD (solid line) and UV-vis (dashed line) spectra of **apy·(*S*,*S*)-2** measured in KBr disk.

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