Supplimenatry Informations

Fluorescence turn-on Sensor for HSO_4^-

Hyun Jung Kim,[1] Sankarprasad Bhuniya,[1] Rakesh Kumar Mahajan,[2,]* Rajiv Puri,[2] Hongguang Liu,[3] Kyoung Chul Ko,[3] Jin Yong Lee,[3,]* and Jong Seung Kim[1,]*

[1]*Department of Chemistry, Korea University, Seoul 136-701, Korea.*
[2]*Department of Chemistry, Guru Nanak Dev University, Amritsar 143005, India*
[3]*Institute of Basic Science, Sungkyunkwan University, Suwon 440-746, Korea.*

* Corresponding authors: rakesh_chem@yahoo.com (R. K. Mahajan), jinylee@skku.edu (J. Y. Lee), jongskim@korea.ac.kr (J. S. Kim)

Table of contents

1. Instruments and reagents

All fluorescence and UV-vis absorption spectra were recorded in RF-5301PC and S-3100 spectrophotometer, respectively. NMR and mass spectra were recorded at Varian instrument (300 MHz) and JMS-700 MStation mass spectrometer, respectively. 1-Aminopyrene, 7-amino-4-(trifluoromethyl)coumarin, 2-hydroxybenzaldehyde, benzaldehyde, and all anionic compounds such as TBA^+ salts of F^-, Cl^-, Br^-, I^-, $CH_3CO_2^-$, HSO_4^-, $H_2PO_4^-$, NO_3^-, and OH^- were purchased from Aldrich and used as received. All solvents were analytical reagents and from Duksan Pure Chemical Co., Ltd. CH_3CN for spectra detection was HPLC reagent without fluorescent impurity and H_2O was deionized water.

2. General procedure for fluorescence studies

Fluorescence spectra were recorded with a RF-5301PC spectrofluorophotometer. Stock solutions (1.00 mM) of TBA^+ salts were prepared in H_2O/CH_3CN (1:1, v/v). Stock solutions of free **1** - **3** (0.030 mM) were prepared in H_2O/CH_3CN (1:1, v/v). Excitations were carried out at 355 nm (for **1** - **3**) with all excitation slit widths is 3 nm, that of emission is 3 nm. Titration experiments were performed with 3.0 μM solutions of **1** in H_2O/CH_3CN (1:1, v/v) and various concentrations of metal perchlorates in H_2O/CH_3CN (1:1, v/v). From the calculated concentrations of the free ligands and complexed forms of **1** in the fluorescence titration experiments, association constants were obtained with the computer program ENZFITTER.

3. Job plot experiment

For the Job plot experiment, **1** (10.0 μM) in H_2O/CH_3CN (1:1, v/v) and tetrabutylammonium hydrogensulfate (10.0 μM) in H_2O/CH_3CN (1:1, v/v) were prepared as stock solutions. The concentrations of each CH_3CN solution were varied, but the total volume was fixed at 4.0 mL. After the mixture was shaken, the absorbance intensity at 355 nm was recorded.

4. ISE membrane preparation

The general procedure adopted to prepare PVC was as follows: Ionophore **1**, PVC, ToMACl, and DOS plasticizer were dissolved in a minimum quantity of freshly distilled THF. The resulting mixture was stirred vigorously and transferred into a 50-mm Petri dish after removing air bubbles. The solvent was allowed to evaporate off at room temperature overnight. A flexible transparent membrane of thickness ~ 0.4 mm was obtained. A membrane of suitable size was cut and fixed to a PVC tube with the help of glue and conditioned in 1.0×10^{-2} M hydrogen sulphate ion solution for 2-3 days.

5. Supplementary spectral data

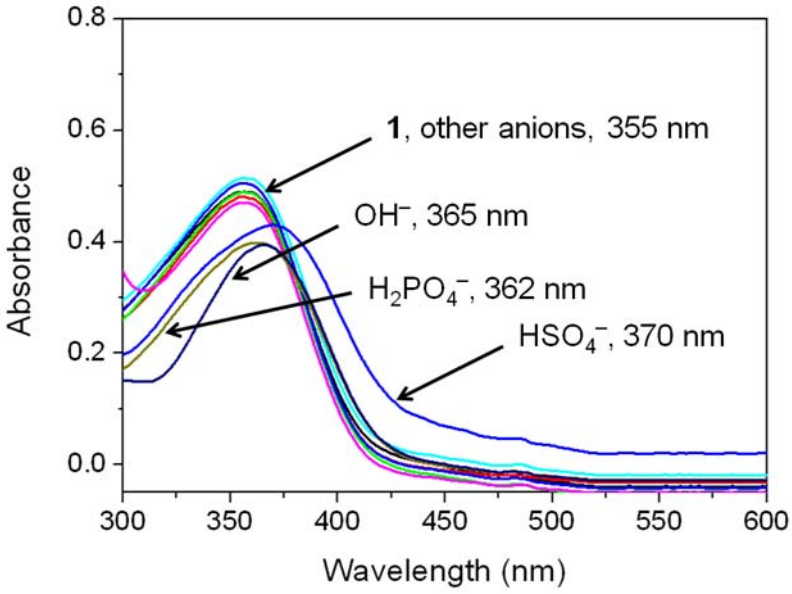


Figure S1. UV spectra of **1** (10.0 μM) upon addition of TBA^+ salts of F^-, Cl^-, Br^-, I^-, $CH_3CO_2^-$, HSO_4^-, $H_2PO_4^-$, NO_3^-, and OH^- (100 equiv) in 1:1 CH_3CN/H_2O (v/v) solution.

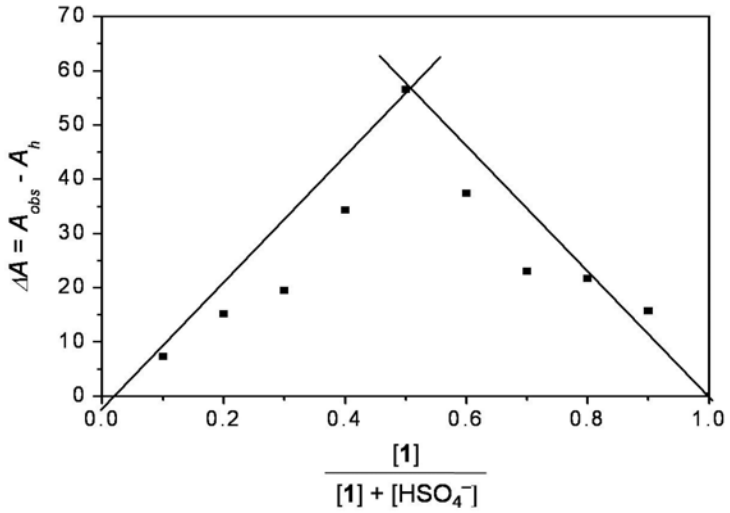


Figure S2. Job's plot for a 1:1 complex of **1** and HSO_4^-, where the difference in absorbance intensity at 355 nm is plotted against the mole fraction of **1** at an invariant total concentration of 3.0 μM in 1:1 CH_3CN/H_2O (v/v) solution.

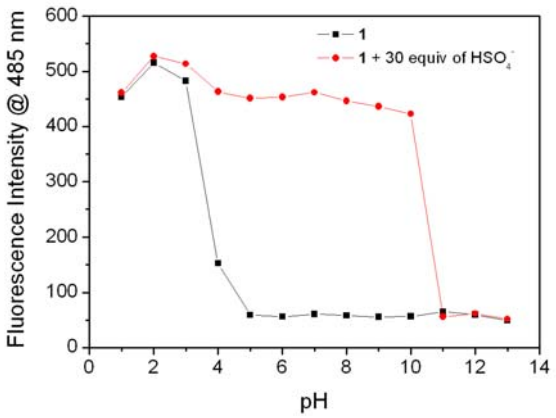


Figure S3. Fluorescence intensity at 485 nm (excitation at 355 nm) of **1** (3.0 μM) and **1** + 30 equiv of HSO_4^- in 1:1 CH_3CN/H_2O (v/v) solution with different pH conditions.

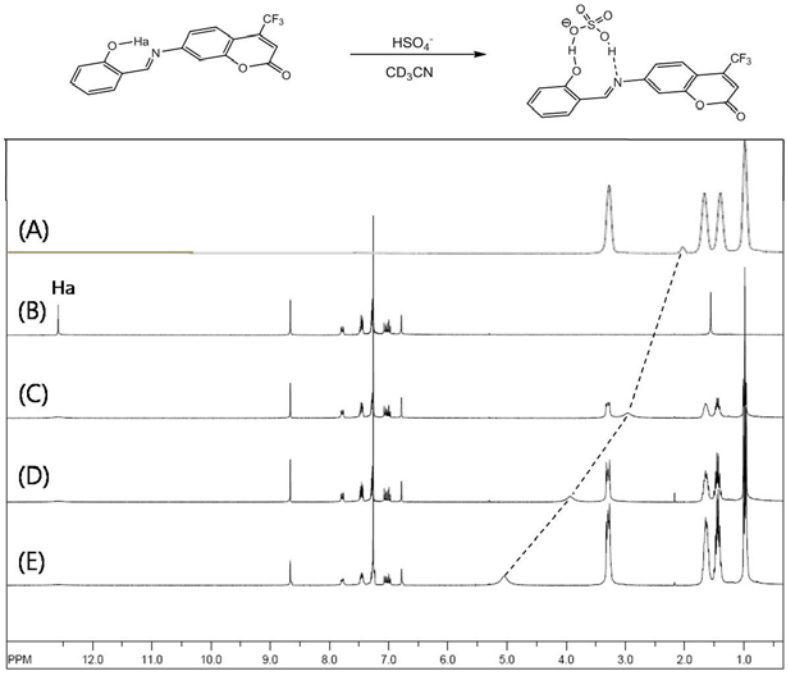


Figure S4. ^1H NMR data of $TBA^+HSO_4^-$ (A), ^1H NMR data of **1** (5 mM) in CD_3CN in the absence (B) and presence of 0.5 eq (C) 1 eq (D) 2 eq (E) of $TBA^+HSO_4^-$.

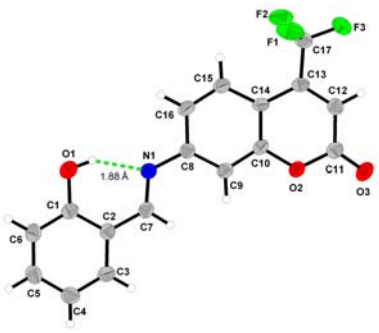


Figure S5. X-ray crystal structure of **1** with hydrogen bond represented by dotted bond

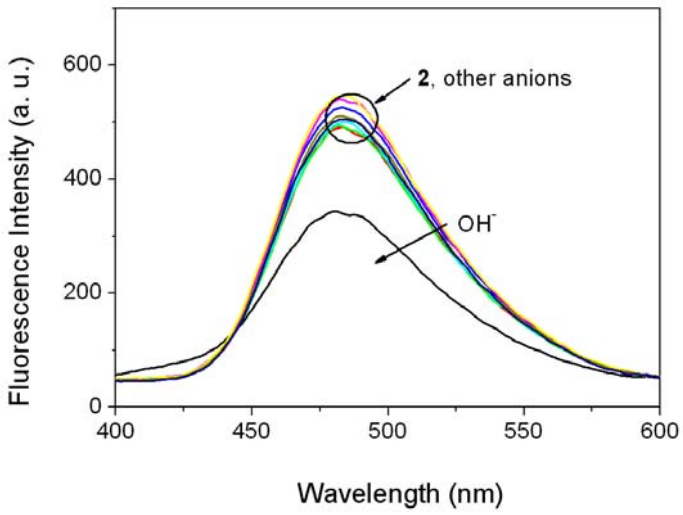


Figure S6. Fluorescence spectra of **2** (3.0 μM) λ_{ex}=355 nm upon addition of TBA^+ salts of F^-, Cl^-, Br^-, I^-, $CH_3CO_2^-$, HSO_4^-, $H_2PO_4^-$, NO_3^-, and OH^- (100 equiv) in 1:1 CH_3CN/H_2O (v/v) solution.

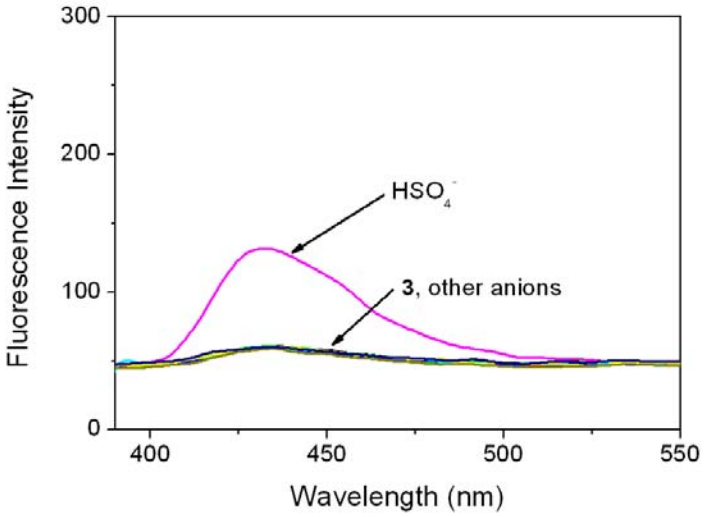


Figure S7. Fluorescence spectra of **3** (3.0 μM) λ_{ex}=375 nm upon addition of TBA^+ salts of F^-, Cl^-, Br^-, I^-, $CH_3CO_2^-$, HSO_4^-, $H_2PO_4^-$, NO_3^-, and OH^- (100 equiv) in 1:1 CH_3CN/H_2O (v/v) solution.

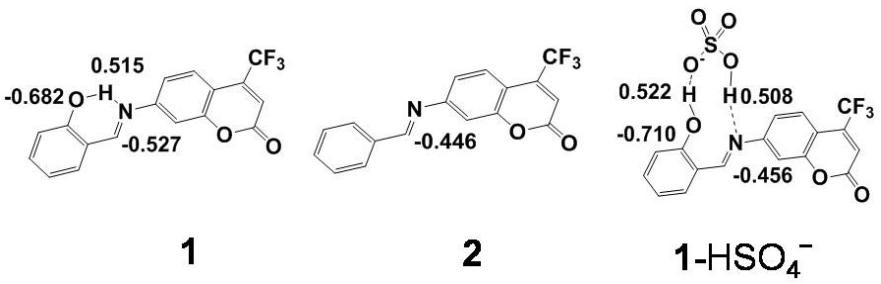


Figure S8. Calculated NBO charges of **1**, **2** and **1**-HSO_4^-.

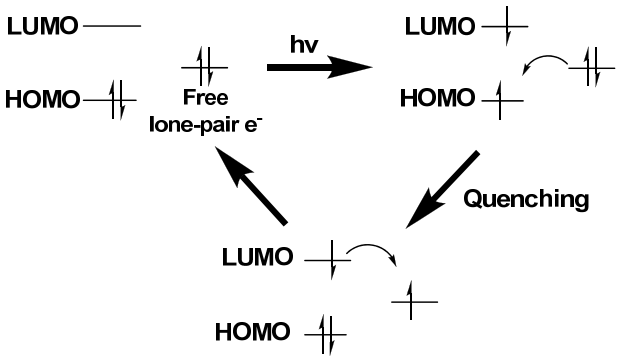


Figure S9. Mechanism for PET.

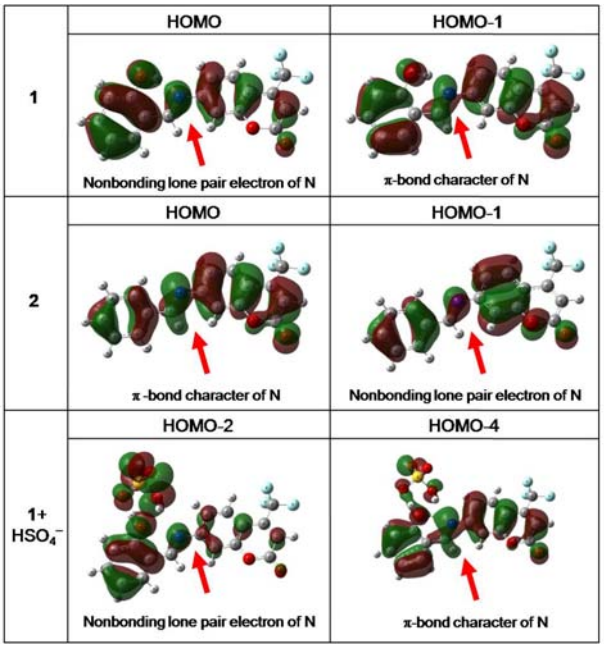


Figure S10. Calculated Molecular orbitals of **1, 2** and **1**-HSO_4^-.

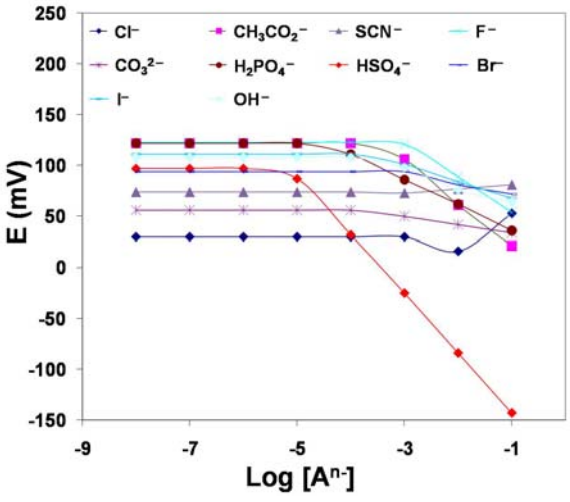


Figure S11. Potentiometric response curves of ISEs based on receptor **1** towards different anions.

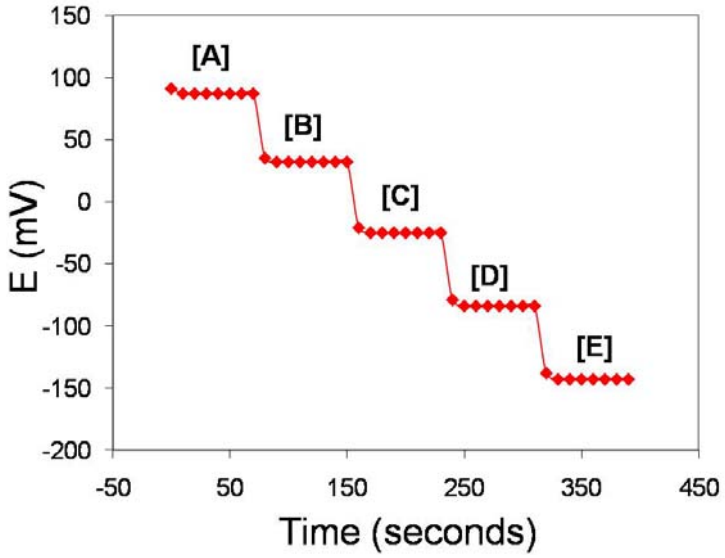


Figure S12. Time response curve of ion selective electrode based on compound **1** at various concentrations of HSO_4^- ions. [A] 1.0×10^{-5} M [B] 1.0×10^{-4} M [C] 1.0×10^{-3} M [D] 1.0×10^{-2} M [E] 1.0×10^{-1} M.

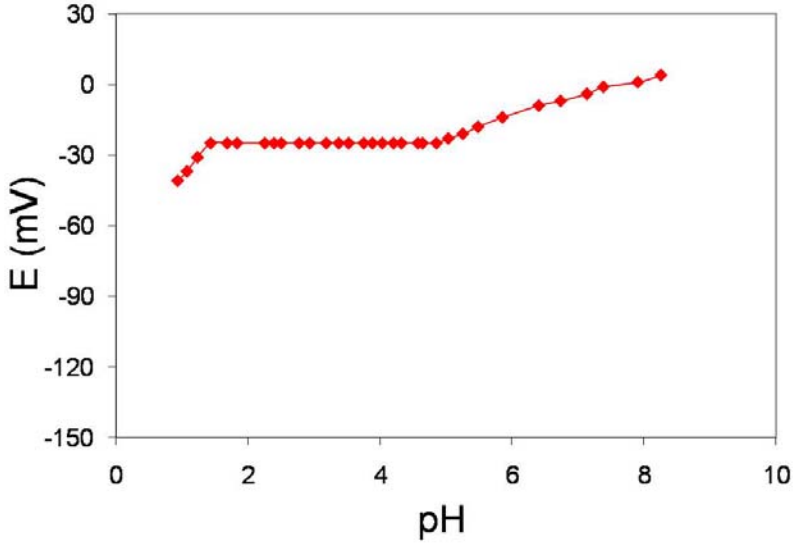


Figure S13. Effect of pH on potential response of HSO_4^- ion selective electrode based on receptor **1.**

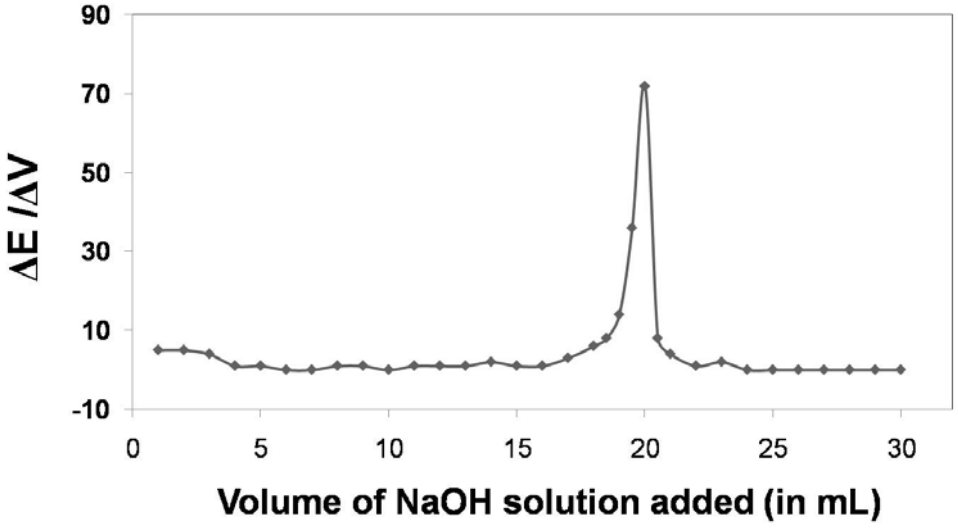
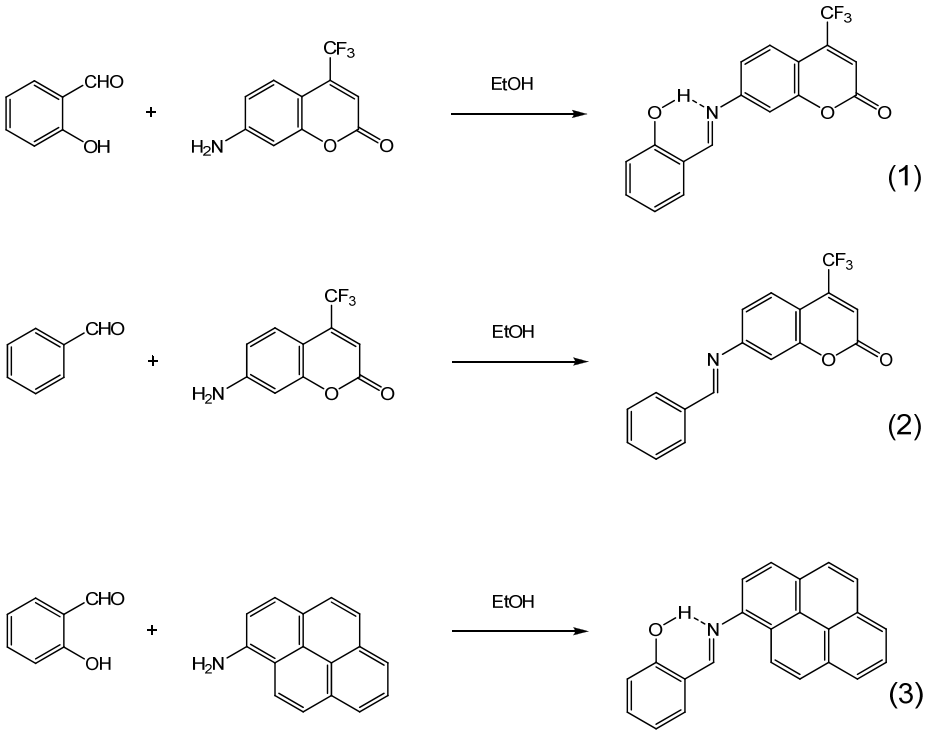
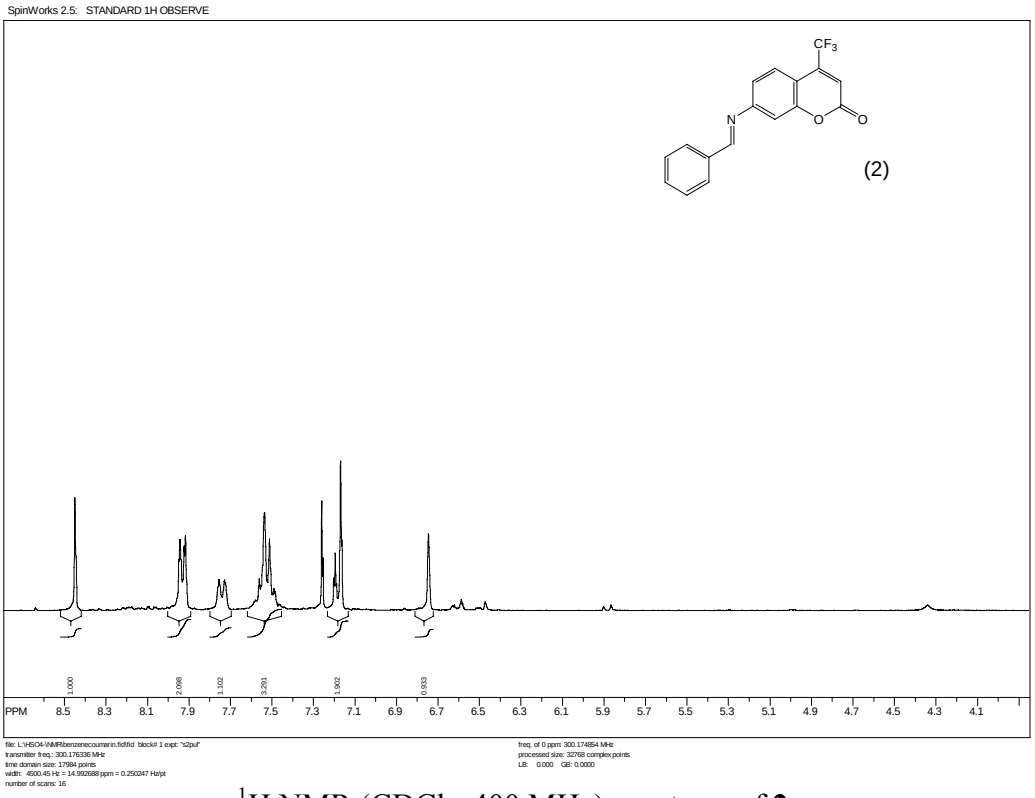


Figure S14. Derivative curve for the titration of 1.0×10^{-2} M HSO_4^- solution with 1.0×10^{-2} M NaOH solution.

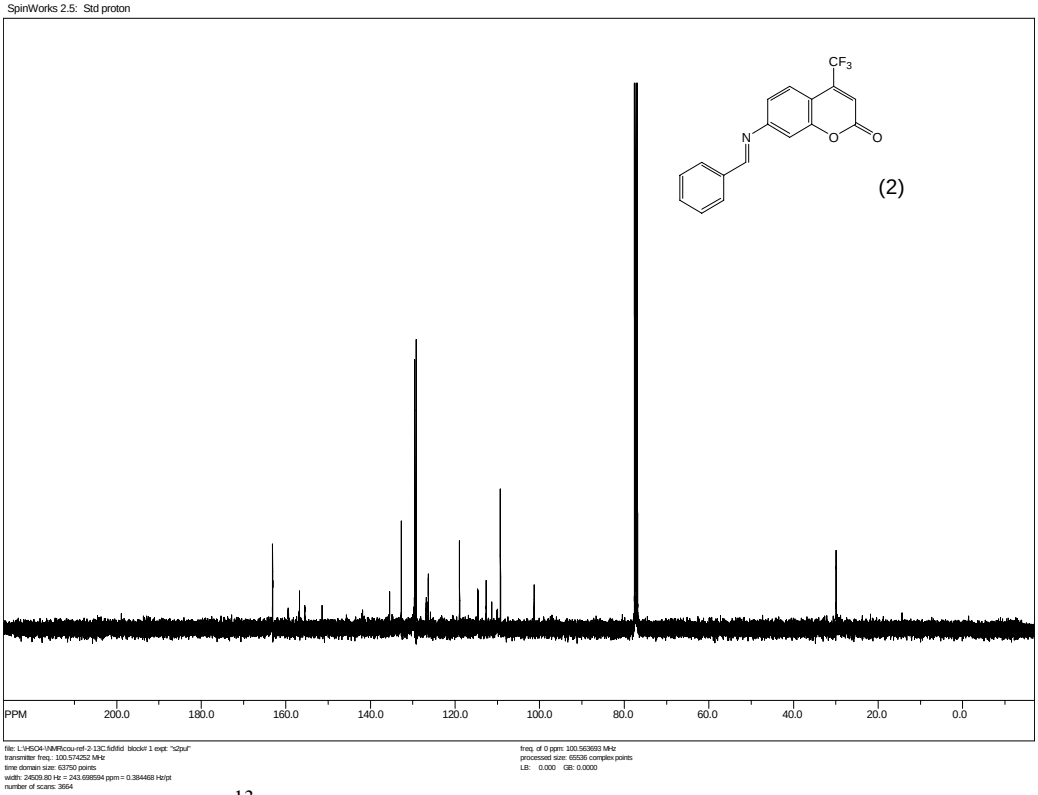


Scheme S1. Synthetic pathway of **1-3**.

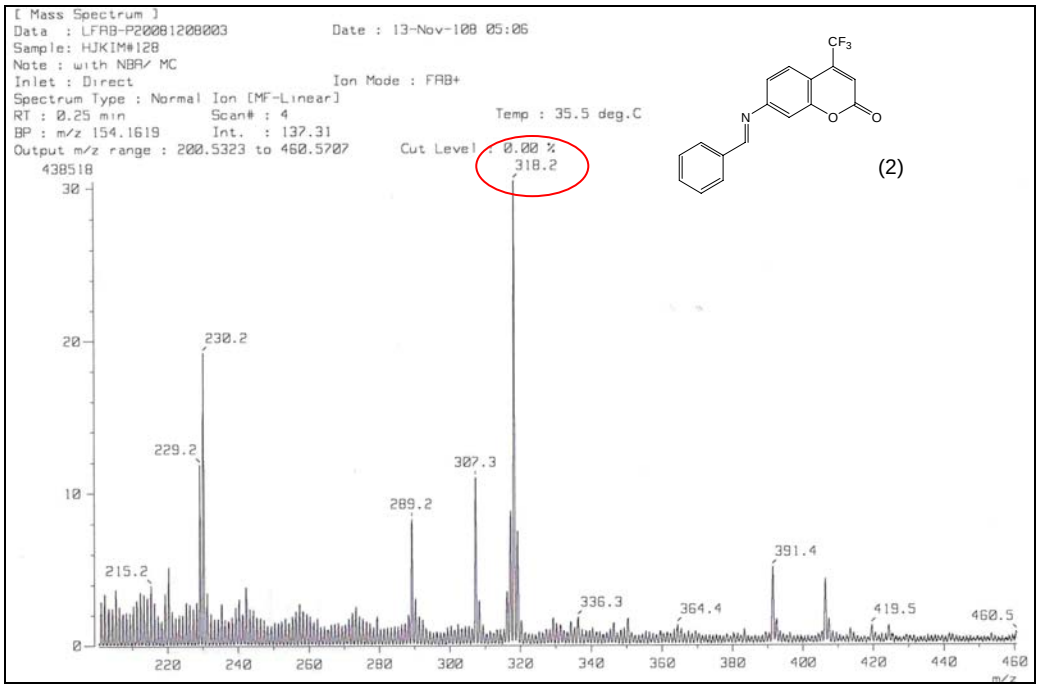
6. NMR copies



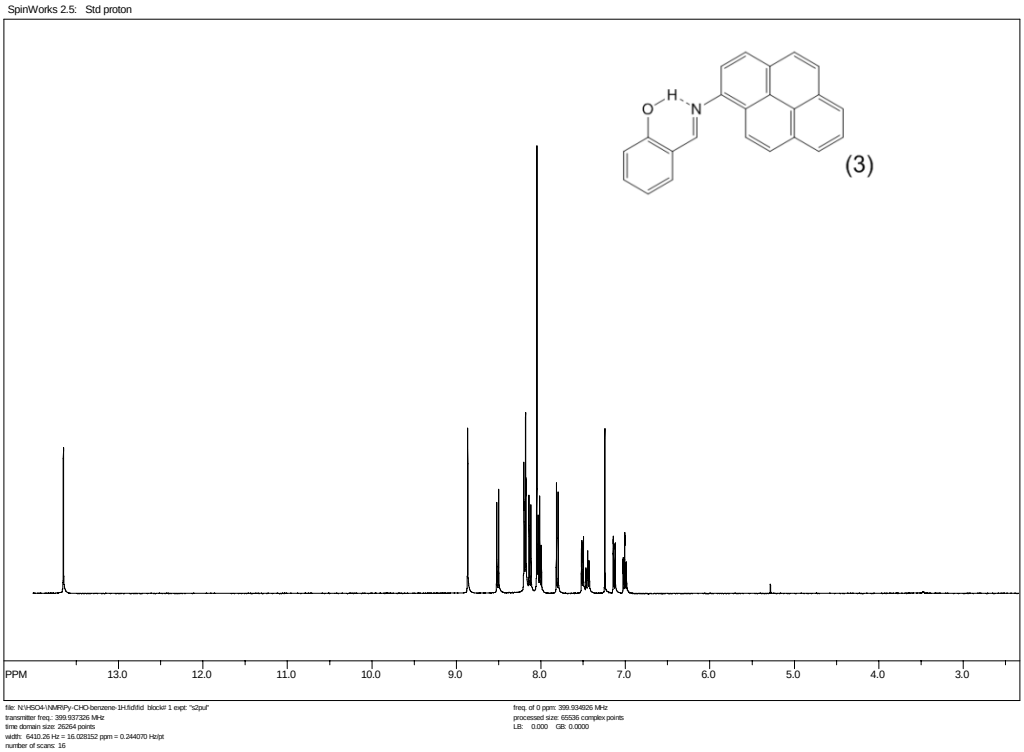
^{1}H NMR ($CDCl_3$, 400 MHz) spectrum of **2**.



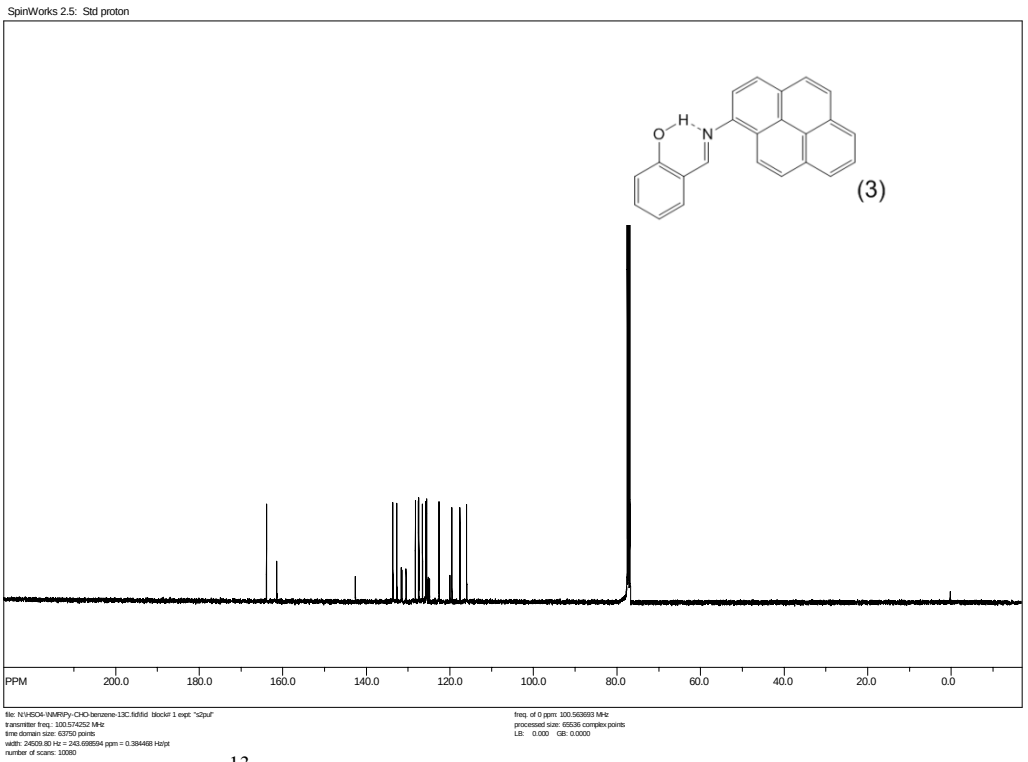
^{13}C NMR ($CDCl_3$, 100 MHz) spectrum of **2**.



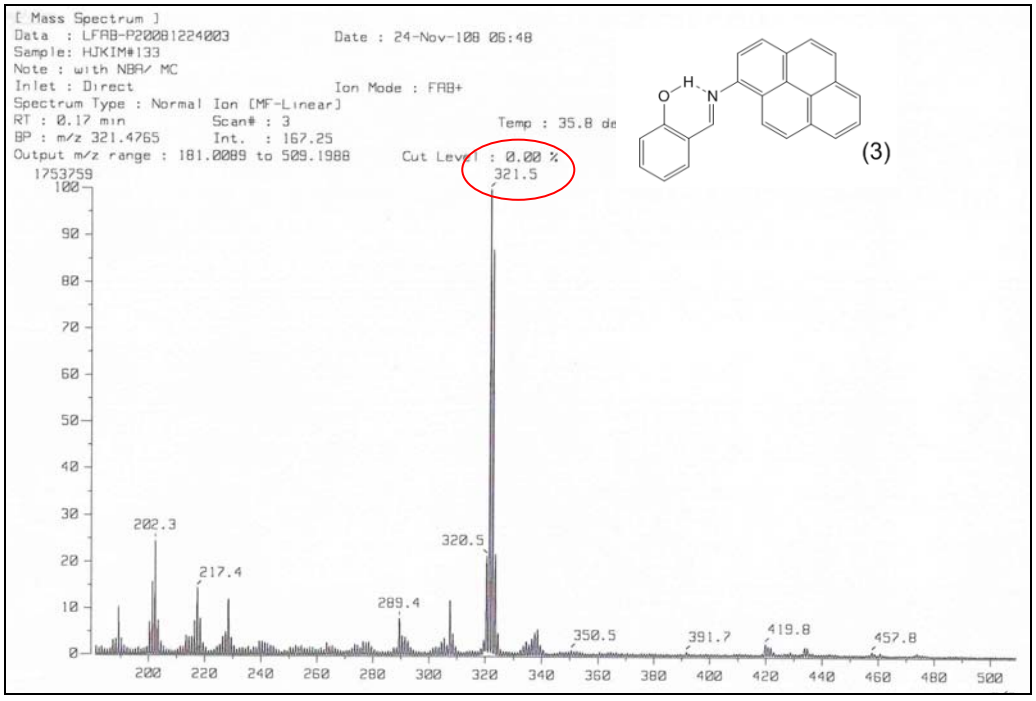
FAB-Mass spectrum of **2**.



^{1}H NMR ($CDCl_3$, 400 MHz) spectrum of **3**.



^{13}C NMR ($CDCl_3$, 100 MHz) spectrum of **3**.



FAB-Mass spectrum of **3**.

7. Experimental Section

***Synthesis of 1*:** A portion of salicylaldehyde (0.58 g, 4.37 mmol) and 7-amino-4-(trifluoromethyl) coumarin (1 g, 4.37 mmol) were combined in hot absolute ethanol (50.0 mL) for 2h to obtain the orange solid of **1** (1.2 g, 2.52 mmol) in 82 % yield.

***Synthesis of 2*:** A portion of benzaldehyde (443 μl, 4.37 mmol) and 7-amino-4-(trifluoromethyl)coumarin (1 g, 4.37 mmol) were combined in hot absolute ethanol (50.0 mL) for 2h to obtain the yellow oil of **2** (0.8 g, 2.52 mmol) in 58 % yield. IR (KBr pellet, cm^{-1}): 1698, 1616, 603; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.74 (s, 1H), 7.16 (s, 1H), 7.20 (d, 1H), 7.48-7.56 (m, 3H), 7.72 (d, J = 9.3Hz, 1H), 7.91 (d, 2H), 8.44 (s, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 163.1, 160.0, 156.8, 135.5, 132.7, 129.5, 129.2, 126.3, 118.9, 114.6, 112.6, 109.2, 101.3, 29.9 ppm. FAB MS *m/z* (M^+): calcd, 317.26. Found, 318.20.

***Synthesis of 3*:** A portion of salicylaldehyde (0.05 g, 0.46 mmol) and 1-aminopyrene (0.1 g, 0.46 mmol) were combined in hot absolute ethanol (10.0 mL) for 2h to obtain the greenish-yellow oil of **3** (0.1 g, 2.52 mmol) in 68 % yield. IR (KBr pellet, cm^{-1}): 1687, 1624; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.98 (t, 1H), 7.12 (d, 1H), 7.42 (t, 1H), 7.49 (d, 1H), 7.79 (d, 1H), 7.99 (t, 1H), 8.04 (s, 1H), 8.11 (d, 1H), 8.18 (d, 1H), 8.49 (d, 1H), 8.86 (s, 1H), 8.04 (s, 1H), 13.65 (s, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 163.8, 161.4, 142.6, 133.6, 132.7, 131.6, 130.4, 128.2, 127.4, 126.5, 125.8, 125.6, 125.5, 125.4, 125.1, 124.9, 122.6, 119.9, 119.5, 117.5, 115.9 ppm. FAB MS *m/z* (M^+): calcd, 321.37. Found, 321.50.

8. Crystallographic Data

Crystal data and structure refinement for yk982sadP21n.

Empirical formula	C17 H10 F3 N O3	
Formula weight	333.26	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 9.855(2) Å	= 90.00°
	b = 5.4280(11) Å	= 91.02(3)°
	c = 26.716(5) Å	= 90.00°
Volume	1428.9(5) $Å^3$	
Z	4	
Density (calculated)	1.549 Mg/m^3	
Absorption coefficient	0.133 mm^{-1}	
F(000)	680	
Crystal size	0.30 x 0.03 x 0.03 mm^3	
Theta range for data collection	2.19 to 26.00 .	
Index ranges	$-9 \le h \le 12$, $-6 \le k \le 6$, $-32 \le l \le 30$	
Reflections collected	7321	
Independent reflections	2796 [R(int) = 0.0552]	
Completeness to theta = 26.00	99.5 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2796 / 0 / 219	
Goodness-of-fit on F^2	0.925	
Final R indices [I>2sigma(I)]	R1 = 0.0530, wR2 = 0.1193	
R indices (all data)	R1 = 0.1446, wR2 = 0.1541	
Extinction coefficient	0.0079(19)	
Largest diff. peak and hole	0.192 and -0.236 $e.Å^{-3}$	

Atomic coordinates (Å x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for yk982sadP21n. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2662(3)	5352(6)	8681(1)	52(1)
C(2)	1860(3)	3243(6)	8736(1)	45(1)
C(3)	958(3)	2623(6)	8348(1)	55(1)
C(4)	833(4)	4031(7)	7924(1)	62(1)
C(5)	1629(4)	6110(7)	7879(1)	62(1)
C(6)	2532(4)	6779(6)	8251(1)	61(1)
C(7)	1955(3)	1722(6)	9175(1)	50(1)
C(8)	2791(3)	728(6)	9979(1)	46(1)
C(9)	1948(3)	-1264(6)	10058(1)	49(1)
C(10)	2057(3)	-2512(5)	10505(1)	45(1)
C(11)	1197(3)	-5930(7)	10973(1)	51(1)
C(12)	2084(3)	-5187(6)	11379(1)	52(1)
C(13)	2927(3)	-3258(6)	11346(1)	46(1)
C(14)	2982(3)	-1864(5)	10887(1)	43(1)
C(15)	3836(3)	146(6)	10791(1)	50(1)
C(16)	3736(3)	1392(6)	10349(1)	52(1)
C(17)	3748(4)	-2479(7)	11793(1)	57(1)
F(1)	3364(2)	-300(4)	11958(1)	94(1)
F(2)	5052(2)	-2255(4)	11708(1)	85(1)
F(3)	3641(3)	-4007(5)	12175(1)	104(1)
N(1)	2749(3)	2233(5)	9546(1)	51(1)
O(1)	3547(3)	6062(5)	9042(1)	73(1)
O(2)	1185(2)	-4477(4)	10554(1)	53(1)
O(3)	466(2)	-7702(4)	10973(1)	64(1)

Bond lengths [Å] and angles [°] for yk982sadP21n.

C(1)-O(1)	1.345(4)
C(1)-C(6)	1.389(4)
C(1)-C(2)	1.400(4)
C(2)-C(3)	1.396(4)
C(2)-C(7)	1.436(4)
C(3)-C(4)	1.371(4)
C(3)-H(3)	0.9300
C(4)-C(5)	1.380(5)
C(4)-H(4)	0.9300
C(5)-C(6)	1.372(4)
C(5)-H(5)	0.9300
C(6)-H(6)	0.9300
C(7)-N(1)	1.281(4)
C(7)-H(7)	0.9300
C(8)-C(9)	1.382(4)
C(8)-C(16)	1.393(4)
C(8)-N(1)	1.418(4)
C(9)-C(10)	1.374(4)
C(9)-H(9)	0.9300
C(10)-O(2)	1.378(3)
C(10)-C(14)	1.402(4)
C(11)-O(3)	1.202(3)
C(11)-O(2)	1.368(4)
C(11)-C(12)	1.440(4)
C(12)-C(13)	1.341(4)
C(12)-H(12)	0.9300
C(13)-C(14)	1.442(4)
C(13)-C(17)	1.493(4)
C(14)-C(15)	1.405(4)
C(15)-C(16)	1.363(4)
C(15)-H(15)	0.9300
C(16)-H(16)	0.9300
C(17)-F(2)	1.315(4)
C(17)-F(1)	1.320(4)
C(17)-F(3)	1.320(4)
O(1)-H(1)	0.8200
O(1)-C(1)-C(6)	118.8(3)

O(1)-C(1)-C(2)	121.3(3)
C(6)-C(1)-C(2)	120.0(3)
C(3)-C(2)-C(1)	118.2(3)
C(3)-C(2)-C(7)	120.0(3)
C(1)-C(2)-C(7)	121.8(3)
C(4)-C(3)-C(2)	121.8(3)
C(4)-C(3)-H(3)	119.1
C(2)-C(3)-H(3)	119.1
C(3)-C(4)-C(5)	119.0(3)
C(3)-C(4)-H(4)	120.5
C(5)-C(4)-H(4)	120.5
C(6)-C(5)-C(4)	121.0(3)
C(6)-C(5)-H(5)	119.5
C(4)-C(5)-H(5)	119.5
C(5)-C(6)-C(1)	120.0(3)
C(5)-C(6)-H(6)	120.0
C(1)-C(6)-H(6)	120.0
N(1)-C(7)-C(2)	122.5(3)
N(1)-C(7)-H(7)	118.8
C(2)-C(7)-H(7)	118.8
C(9)-C(8)-C(16)	119.3(3)
C(9)-C(8)-N(1)	124.6(3)
C(16)-C(8)-N(1)	116.1(3)
C(10)-C(9)-C(8)	118.7(3)
C(10)-C(9)-H(9)	120.6
C(8)-C(9)-H(9)	120.6
C(9)-C(10)-O(2)	115.2(3)
C(9)-C(10)-C(14)	123.3(3)
O(2)-C(10)-C(14)	121.5(3)
O(3)-C(11)-O(2)	117.7(3)
O(3)-C(11)-C(12)	125.4(3)
O(2)-C(11)-C(12)	116.9(3)
C(13)-C(12)-C(11)	122.4(3)
C(13)-C(12)-H(12)	118.8
C(11)-C(12)-H(12)	118.8
C(12)-C(13)-C(14)	120.0(3)
C(12)-C(13)-C(17)	119.7(3)
C(14)-C(13)-C(17)	120.3(3)
C(10)-C(14)-C(15)	116.5(3)
C(10)-C(14)-C(13)	117.0(3)

C(15)-C(14)-C(13)	126.4(3)
C(16)-C(15)-C(14)	120.5(3)
C(16)-C(15)-H(15)	119.7
C(14)-C(15)-H(15)	119.7
C(15)-C(16)-C(8)	121.6(3)
C(15)-C(16)-H(16)	119.2
C(8)-C(16)-H(16)	119.2
F(2)-C(17)-F(1)	105.2(3)
F(2)-C(17)-F(3)	106.4(3)
F(1)-C(17)-F(3)	106.1(3)
F(2)-C(17)-C(13)	114.0(3)
F(1)-C(17)-C(13)	111.6(3)
F(3)-C(17)-C(13)	113.0(3)
C(7)-N(1)-C(8)	121.0(3)
C(1)-O(1)-H(1)	109.5
C(11)-O(2)-C(10)	121.9(2)

Symmetry transformations used to generate equivalent atoms:

Anisotropic displacement parameters ($Å^2 x\ 10^3$) for yk982sadP21n. 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}
C(1)	54(2)	43(2)	58(2)	-5(2)	-1(2)	-2(2)
C(2)	47(2)	36(2)	54(2)	2(2)	0(2)	4(2)
C(3)	50(2)	50(2)	65(2)	0(2)	-10(2)	-3(2)
C(4)	63(2)	57(2)	64(2)	2(2)	-11(2)	4(2)
C(5)	73(2)	52(2)	60(2)	12(2)	0(2)	2(2)
C(6)	74(2)	42(2)	65(2)	7(2)	-2(2)	-6(2)
C(7)	50(2)	37(2)	62(2)	4(2)	-3(2)	-6(2)
C(8)	48(2)	42(2)	49(2)	-1(2)	-1(2)	-2(2)
C(9)	51(2)	41(2)	53(2)	-2(2)	-9(2)	-7(2)
C(10)	42(2)	38(2)	56(2)	-3(2)	0(2)	-6(2)
C(11)	47(2)	43(2)	61(2)	-3(2)	0(2)	1(2)
C(12)	53(2)	47(2)	55(2)	6(2)	-1(2)	4(2)
C(13)	41(2)	44(2)	53(2)	-7(2)	-7(2)	5(2)
C(14)	41(2)	40(2)	49(2)	-5(2)	-10(2)	2(2)
C(15)	47(2)	47(2)	56(2)	-3(2)	-8(2)	-4(2)
C(16)	47(2)	46(2)	63(2)	-4(2)	-6(2)	-9(2)
C(17)	60(3)	55(2)	57(2)	3(2)	-11(2)	5(2)
F(1)	117(2)	85(2)	80(2)	-34(1)	-28(1)	26(2)
F(2)	54(1)	116(2)	84(2)	-6(1)	-20(1)	-1(1)
F(3)	132(2)	109(2)	69(2)	31(2)	-39(1)	-40(2)
N(1)	54(2)	49(2)	50(2)	2(1)	-7(1)	-8(1)
O(1)	87(2)	61(2)	69(2)	5(1)	-15(2)	-32(1)
O(2)	52(1)	47(1)	59(2)	6(1)	-10(1)	-12(1)
O(3)	60(2)	50(2)	81(2)	0(1)	-1(1)	-17(1)

Hydrogen coordinates (Å x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for yk982sadP21n.

	x	y	z	U(eq)
H(3)	427	1216	8377	66
H(4)	222	3593	7670	74
H(5)	1552	7072	7592	74
H(6)	3058	8188	8216	73
H(7)	1420	314	9191	60
H(9)	1320	-1751	9814	58
H(12)	2071	-6077	11677	62
H(15)	4476	632	11032	60
H(16)	4312	2716	10293	63
H(1)	3494	5128	9282	109

Torsion angles [°] for yk982sadP21n.

O(1)-C(1)-C(2)-C(3)	179.7(3)
C(6)-C(1)-C(2)-C(3)	0.8(5)
O(1)-C(1)-C(2)-C(7)	-0.8(5)
C(6)-C(1)-C(2)-C(7)	-179.6(3)
C(1)-C(2)-C(3)-C(4)	-0.8(5)
C(7)-C(2)-C(3)-C(4)	179.7(3)
C(2)-C(3)-C(4)-C(5)	0.4(5)
C(3)-C(4)-C(5)-C(6)	-0.2(5)
C(4)-C(5)-C(6)-C(1)	0.3(5)
O(1)-C(1)-C(6)-C(5)	-179.5(3)
C(2)-C(1)-C(6)-C(5)	-0.6(5)
C(3)-C(2)-C(7)-N(1)	-178.7(3)
C(1)-C(2)-C(7)-N(1)	1.8(5)
C(16)-C(8)-C(9)-C(10)	1.0(5)
N(1)-C(8)-C(9)-C(10)	-177.4(3)
C(8)-C(9)-C(10)-O(2)	-179.9(3)
C(8)-C(9)-C(10)-C(14)	0.1(5)
O(3)-C(11)-C(12)-C(13)	176.5(3)
O(2)-C(11)-C(12)-C(13)	-4.1(5)
C(11)-C(12)-C(13)-C(14)	-0.8(5)
C(11)-C(12)-C(13)-C(17)	175.7(3)
C(9)-C(10)-C(14)-C(15)	-1.0(5)
O(2)-C(10)-C(14)-C(15)	179.0(3)
C(9)-C(10)-C(14)-C(13)	176.6(3)
O(2)-C(10)-C(14)-C(13)	-3.4(4)
C(12)-C(13)-C(14)-C(10)	4.5(4)
C(17)-C(13)-C(14)-C(10)	-172.0(3)
C(12)-C(13)-C(14)-C(15)	-178.1(3)
C(17)-C(13)-C(14)-C(15)	5.4(5)
C(10)-C(14)-C(15)-C(16)	0.9(4)
C(13)-C(14)-C(15)-C(16)	-176.5(3)
C(14)-C(15)-C(16)-C(8)	0.1(5)
C(9)-C(8)-C(16)-C(15)	-1.1(5)
N(1)-C(8)-C(16)-C(15)	177.4(3)
C(12)-C(13)-C(17)-F(2)	128.3(3)
C(14)-C(13)-C(17)-F(2)	-55.2(4)
C(12)-C(13)-C(17)-F(1)	-112.8(4)
C(14)-C(13)-C(17)-F(1)	63.7(4)

C(12)-C(13)-C(17)-F(3)	6.6(5)
C(14)-C(13)-C(17)-F(3)	-176.8(3)
C(2)-C(7)-N(1)-C(8)	178.3(3)
C(9)-C(8)-N(1)-C(7)	-4.5(5)
C(16)-C(8)-N(1)-C(7)	177.1(3)
O(3)-C(11)-O(2)-C(10)	-175.3(3)
C(12)-C(11)-O(2)-C(10)	5.3(4)
C(9)-C(10)-O(2)-C(11)	178.4(3)
C(14)-C(10)-O(2)-C(11)	-1.6(4)

Symmetry transformations used to generate equivalent atoms:

Hydrogen bonds for yk982sadP21n.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(12)-H(12)...F(1)#1	0.93	2.72	3.409(4)	131.3
C(4)-H(4)...F(1)#2	0.93	2.77	3.580(4)	145.6
C(5)-H(5)...F(2)#2	0.93	2.76	3.522(4)	139.4
C(12)-H(12)...F(3)	0.93	2.31	2.677(4)	102.7
C(15)-H(15)...F(2)	0.93	2.45	3.005(4)	118.3
C(15)-H(15)...F(1)	0.93	2.77	3.169(4)	106.9
O(1)-H(1)...N(1)	0.82	1.88	2.606(3)	147.4

Symmetry transformations used to generate equivalent atoms:

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