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SUPPORTING INFORMATION for

S-(1-Oxido-2-pyridinyl) 1,1,3,3-Tetramethylthiouronium Hexafluorophosphate (HOTT). A New Reagent for Preparing Hindered Barton Esters.

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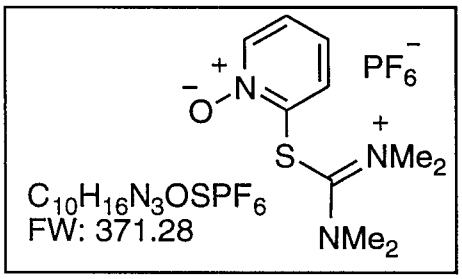
General Details. All reactions were performed under an inert, moisture-free atmosphere (N_2 or Ar). Solvents/reagents were purified beyond reagent grade as follows: THF was distilled from Na/benzophenone; CH_2Cl_2 and tertiary amines were distilled from CaH_2 (0-1 mm grain size, ACROS). All distillations were done in an inert (N_2 or Ar), dry environment. Tert. dodecanethiol (mixture of isomers, Aldrich) and 2-mercaptopyridine-N-oxide (Aldrich) were used as received. Commercial synthetic *N*-acetylneuraminic acid (Aldrich) was peracetylated according to Meindl and Tuppy.[1] 2,3:4,6-Di-O-isopropylidene-2-keto-L-gulonic acid monohydrate (Aldrich) was dehydrated by azeotropic distillation using benzene as described by Crich.[2] Compound **13** was synthesized as described previously.[3] *N*-Fmoc-2-Aminoisobutyric acid (Novabiochem) was used as received. Analytical TLC was performed on JT-Baker Si250F plates (0.25 mm). Preparative TLC was performed on E. Merck silica gel 60 F_{254} plates (0.5 mm). TLC plates were visualized by UV illumination followed by charring with either (A) 5% anisaldehyde in (95:5:1) EtOH-AcOH-H_2SO_4, (B) Verghn's reagent (12.5g Ammonium molybdate

and 0.5g Ceric sulfate dissolved in 250 mL of 10% aq. H_2SO_4), or (C) a 0.3% (w/v) ninhydrin solution in (97:3) EtOH-AcOH. ^1H NMR assignments are based on selective homonuclear decoupling experiments and ^{13}C NMR assignments are based on HETCOR experiments. High resolution MS data are reported in units of m/e for M^+ or the highest mass fragment derived from M^+.

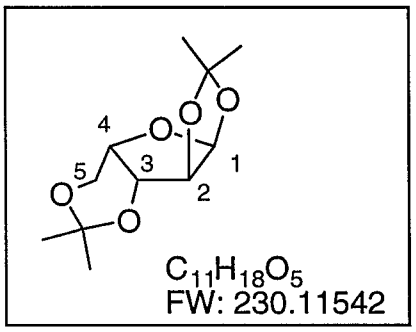


Reagent Preparation: A 3-neck 250 mL round bottom flask was charged with N-hydroxypyridine-2-thione (5.2 g, 0.041 mol) and tetramethylchloroformamidinium hexafluorophosphate[4] (11.4 g, 0.041 mol). The contents of the flask were dissolved in CH_2Cl_2 (100 mL, freshly distilled from CaH_2) under Ar. To this solution was added Et_3N (5.7 mL, 0.041 mol, distilled from CaH_2) slowly over a period of 3-4 min during which time white smoke evolved and the flask became slightly warm. The reaction mixture was stirred for 10 min at which time the solvent was removed on a rotary evaporator. The crude solid product was washed with $CHCl_3$ (2 x 100 mL), suction-filtered (washing with ca. 20 mL of $CHCl_3$), and dried over P_2O_5 under vacuum overnight to give **8** a white powder (77% yield). An analytical sample was prepared by recrystallization from CH_2Cl_2. mp 115-116 °C; ^1H NMR (CD_3CN) δ 8.29-8.31 (m, H-6), 7.73-7.69 (m, H-3), 7.49-7.38 (overlapping m, H-4 & 5), 3.18 (s, 4 x Me); ^{13}C NMR (CD_3CN) δ 200.4 (C=N), 169.6 (C-6), 159.8 (C-3), 156.6 (C-4 or C-5), 156.2 (C-5 or C-4), 146.8 (C-2), 72.8 (Me); Anal. Calcd for $C_{10}H_{16}N_3OSPF_6$: C, 32.34; H, 4.35; N, 11.32. Found: C, 32.10; H, 4.26; N, 11.22.

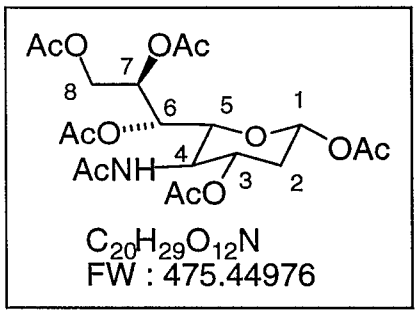
Barton Esterification/Radical Trapping: A solution of carboxylic acid (0.1 M), Et_3N or Hünig's base (4.0 equiv), and DMAP (0.1 equiv) in dry THF was added to a 2-neck flask containing HOTT (1.5 equiv) in the dark under an Ar atmosphere. This mixture was stirred in the dark for the indicated period at which time the reaction was judged to be complete by IR spectroscopy. When IR monitoring was not possible (because of overlapping carbonyl bands), the reaction was allowed to proceed for an arbitrary period of time and presumed to be complete if no starting acid was detected after the trapping step. In general, IR monitoring was performed by injecting 75 μL of the reaction mixture into an IR solution cell (NaCl, 0.1mm gap) in the dark (very dimly lit room). A second solution cell containing THF was

used as a reference. The bright yellow Barton ester solution was then submitted to one of the following trapping reactions: (a) t-dodecanethiol (2 equiv) was added and the mixture was either refluxed or photolyzed (275 W sunlamp) at 0 °C, (b) camphorsulfonic acid (2.1 equiv) was added followed by 2-nitropropene (3 equiv) and the mixture was photolyzed (Brinkmann® Q-Beam® MaxMillion™ spotlight)[5], (c) methyl acrylate (3 equiv) was added and the mixture was photolyzed (Brinkmann spotlight). When the trapping reaction was judged to be complete (TLC), the mixture was submitted to standard extractive workup and the crude product purified by flash chromatography unless otherwise indicated. In the case of trapping reaction (b), a soln of the crude adduct in THF (ca. 0.1 M) was treated with freshly prepared 15% aq $TiCl_3$ (21 equiv, buffered to pH 5-6 w/ NH_4OAc according to McMurry and Melton.[6]). The resulting dark-blue suspension was stirred at 45° C overnight (ca. 12 hr, the blue color fades as the Ti(III) is consumed) under Ar, then worked-up and purified as described.

Characterization Data:

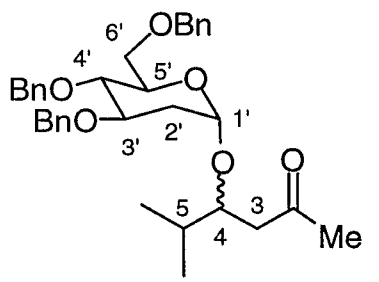


Compound **10**:[2] 78% yield; R_f 0.31, 20:7 hexs-Et_2O (char A); ^1H NMR (300 MHz, $CDCl_3$) δ 6.00 (d, J = 3.7 Hz, H-1), 4.52 (d, J = 3.8 Hz, H-2), 4.29 (d, J = 2.3 Hz, H-4), 4.10-4.07 (m, H-5a & H-5b), 4.03 (br d, J = 1.4 Hz, H-3), 1.49 (s, Me), 1.45 (s, Me), 1.38 (s, Me), 1.32 (s, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 111.4, 105.0 (C-1), 97.3, 84.5 (C-2), 73.0 (C-4), 71.4 (C-3), 60.0 (C-5), 28.7, 26.6, 26.0, & 18.5 (4x Me).

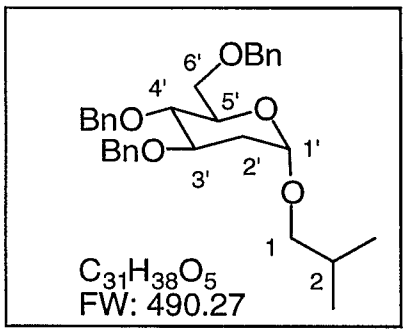


Compound **12**:[7] In this case, the THF was evaporated under reduced pressure and the resulting oil was filtered through silica gel eluting with EtOAc to give crude **12** which was purified by semi-preparative reverse phase HPLC (column: Dynamax-60A, C-18-83-221C; solvent: 15% MeCN/H_2O; flow rate: 18 mL/min; t_R = 31 min). 50% yield; R_f 0.47, 9:1 EtOAc-THF (char C); ^1H NMR (300 MHz, $CDCl_3$) δ 5.65 (dd, $J_{1,2ax}$ = 10.2 Hz, $J_{1,2eq}$ = 1.7 Hz, H-1)5.39 (dd, $J_{6,7}$ = 7.4 Hz, $J_{6,5}$ = 1.9 Hz, H-6), 5.02-5.18 (m, H-7,

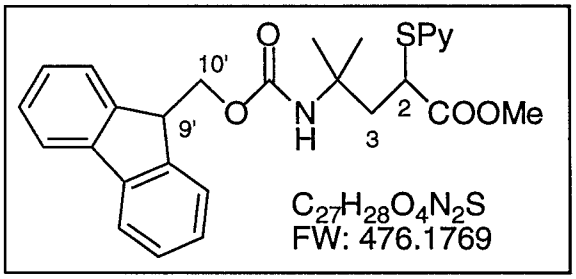
H-3, NH), 4.39 (dd, $J_{8',8}$ = 12.2 Hz, $J_{8',7}$ = 12.6 Hz, H-8'), 4.00-4.11 (m, H-8 & H-4), 3.76 (dd, $J_{5,4}$ = 10.7 Hz, $J_{5,6}$ = 2.2 Hz, H-5), 2.19-2.23 (m, H-2_{eq}), 2.05, 2.05, 2.105, 2.109, and 2.14 (singlets, 5x *O*-acetyl), 1.96 (broad apparent doublet, H-2_{ax}), 1.91 (s, *N*-acetyl).



Compound **14**:[3] 60% yield; (major diastereomer) R_f 0.47, 3:2 hexs-EtOAc (char B); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35-7.17 (m, 3x Ph), 5.1 (d, J = 3.2 Hz, H-1'), 4.9 (d, J = 10.8 Hz, 1/2 $PhCH_2$), 4.66-4.47 (m, 5/2 $PhCH_2$), 3.96-3.90 (m, H-4), 3.90-3.55 (m, 5 H, H-3', H-4', H-5', 2 x H-6'), 2.6 (dd, J_1 = 16.1 Hz, J_2 = 8.7 Hz, H-3a), 2.4 (dd, J_1 = 16.1 Hz, J_2 = 3.5 Hz, H-3b), 2.14 (s + m, COMe, H-2'α), 1.99 (m, H-5), 1.69 (td, J_1= 2.2 Hz, J_2 = 3.7 Hz, H-2'β), 0.88 (d, J = 6.9 Hz, 1/2 Me_2CH), 0.82 (d, J = 6.9 Hz, 1/2 Me_2CH); ^{13}C NMR (APT) (75 MHz, $CDCl_3$) δ 207.7 (+), 138.7 (+), 138.4 (+), 138.2 (+), 128.3 (-), 128.0 (-), 128.0 (-), 128.0 (-), 127.9 (-), 127.8 (-), 127.8 (-), 127.8 (-), 127.7 (-), 127.6 (-), 127.6 (-), 127.6 (-), 127.5 (-), 98.4 (-), 79.7 (-), 78.4 (-), 77.5 (-), 75.0 (+), 73.4 (+), 71.6 (+), 71.4 (-), 68.9 (+), 43.6 (+), 36.0 (+), 31.7 (-), 18.5 (-), 17.0 (-); HRMS (EI) m/z, calc. for $C_{34}H_{42}O_6$ [M^+-H] 545.29031, obsd. 545.25968.



Compound **15**: 76% yield; R_f 0.43, 4:1 hexs-EtOAc (char B); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.16 (m, 3x Ph), 4.93 (d, J = 2.8 Hz, H-1'), 4.89 (d, J =10.8 Hz 1/2 $PhC\underline{H_2}$), 4.7-4.49 (m, 5H, 5/2 $PhC\underline{H_2}$), 4.0 (ddd, J_1 = 13.8 Hz, J_2 = 8.8 Hz, J_3 = 5.0 Hz, H-3'), 3.81-3.58 (m, 4H, H-4', H-5', 2x H-6'), 3.41-3.35 (dd, J_1 = 9.3 Hz, J_2 = 7.0 Hz, H-1), 3.15-3.10 (dd, J_1 = 9.3 Hz, J_2 = 6.3 Hz, H-1), 2.31-2.25 (dd, J_1 = 13.2 Hz, J_2 = 4.9 Hz, H-$2'_{eq}$), 1.83 (quintet, J = 6.7 Hz, H-2), 1.76-1.67 (td, J_1 = 12.6 Hz, J_2 = 3.5 Hz, H-$2'_{ax}$), 0.89 (d, J = 6.7 Hz, 1/2 Me_2CH), 0.88 (d, J = 6.7 Hz, 1/2 Me_2CH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.7, 138.5, 138.1, 128.2, 127.9, 127.8, 127.5, 127.4, 97.4, 78.3, 77.7, 74.9, 74.0, 73.4, 71.7, 70.6, 68.9, 35.5, 28.3, 19.4, 19.3; HRMS (EI) m/z, calc. for $C_{31}H_{38}O_5$ [MH^+] 491.27975, obsd. 491.01502, calc [M^+-Me_2CHCH_2OH] 416.19876, obsd. 416.19989.



Compound **17**: 77% yield; R_f 0.60, 3:2 EtOAc-hexs (char C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.05 (bs, 1 H), 7.77 (d, J = 7.7 Hz, 2 H), 7.64 (d, J = 7.1 Hz, 2 H), 7.26-7.51 (m, 6 H), 7.12-7.15 (m, 1 H), 6.92 (bs, NH), 4.46-4.63 (m, H-2, H-10'a, H-10'b), 4.25 (t, J = 6 Hz, H-9'), 3.74 (s, CO_2Me), 2.58 (dd, J_1 = 14.6 Hz, J_2 = 9.9 Hz, H-3a), 2.00 (d, J = 14.6 Hz, H-3b), 1.36 (s, 4-Me), 1.35 (s, 4-Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.5, 156.8, 154.8, 149.31, 144.09, 144.06, 141.31, 136.32, 127.47, 126.97, 126.94, 124.94, 124.84, 122.2, 120.1, 119.8, 65.1, 52.9, 52.8, 47.5, 45.9, 41.6, 27.4, 24.2; HRMS (EI) m/z, calc. for $C_{27}H_{28}O_4N_2S$ [M^+] 476.1769, obsd. 476.1768.

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Table 1. Crystal data and structure refinement for $C_{10}H_{16}F_6N_3OPS$.

Empirical formula	$C_{10}H_{16}F_6N_3OPS$	
Formula weight	371.29	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 7.063(4) Å	α= 90°.
	b = 27.659(18) Å	β= 110.45(4)°.
	c = 8.258(4) Å	γ = 90°.
Volume	1511.5(15) $Å^3$	
Z	4	
Density (calculated)	1.632 Mg/m^3	
Absorption coefficient	0.388 mm^{-1}	
F(000)	760	
Crystal size	0.60 x 0.50 x 0.50 mm^3	
Theta range for data collection	2.73 to 25.00°.	
Index ranges	$-1 \le h \le 8$, $-1 \le k \le 32$, $-9 \le l \le 9$	
Reflections collected	3421	
Independent reflections	2614 [R(int) = 0.0412]	
Completeness to theta = 25.00°	98.1 %	
Max. and min. transmission	0.8295 and 0.8004	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2614 / 0 / 199	
Goodness-of-fit on F^2	1.089	
Final R indices [I>2sigma(I)]	R1 = 0.0580, wR2 = 0.1395	
R indices (all data)	R1 = 0.0768, wR2 = 0.1588	
Largest diff. peak and hole	0.548 and -0.380 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{10}H_{16}F_6N_3OPS$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S	309(2)	6812(1)	2727(1)	29(1)
P	4135(2)	5780(1)	7350(1)	26(1)
F(1)	3778(4)	6050(1)	5558(3)	46(1)
F(2)	6357(4)	6010(1)	8115(4)	44(1)
F(3)	3234(4)	6246(1)	7993(4)	45(1)
F(4)	1903(4)	5556(1)	6593(3)	40(1)
F(5)	5034(4)	5325(1)	6692(4)	52(1)
F(6)	4460(5)	5517(1)	9139(3)	55(1)
O	-222(5)	7748(1)	3403(4)	44(1)
N(1)	-1997(5)	7554(1)	2630(4)	28(1)
N(2)	211(5)	5886(1)	1855(4)	28(1)
N(3)	-1511(5)	6075(1)	3736(4)	25(1)
C(1)	-2108(6)	7085(1)	2133(5)	25(1)
C(2)	-3952(6)	6880(2)	1197(5)	28(1)
C(3)	-5708(6)	7143(2)	842(5)	33(1)
C(4)	-5570(7)	7617(2)	1438(6)	35(1)
C(5)	-3719(7)	7818(2)	2305(5)	33(1)
C(6)	-449(6)	6198(1)	2759(5)	24(1)
C(7)	840(7)	6026(2)	399(5)	36(1)
C(8)	557(8)	5372(2)	2331(6)	40(1)
C(9)	-1573(7)	6372(2)	5194(5)	34(1)
C(10)	-2883(7)	5656(2)	3374(6)	38(1)

Table 3. Bond lengths [Å] and angles [°] for $C_{10}H_{16}F_6N_3OPS$.

S-C(1)	1.771(4)	F(6)-P-F(2)	90.63(17)
S-C(6)	1.784(4)	F(5)-P-F(2)	89.77(16)
P-F(6)	1.590(3)	F(1)-P-F(2)	89.75(16)
P-F(5)	1.590(3)	F(4)-P-F(2)	179.34(16)
P-F(1)	1.597(3)	F(6)-P-F(3)	90.28(17)
P-F(4)	1.604(3)	F(5)-P-F(3)	179.13(17)
P-F(2)	1.604(3)	F(1)-P-F(3)	88.80(16)
P-F(3)	1.606(3)	F(4)-P-F(3)	89.45(16)
O-N(1)	1.307(5)	F(2)-P-F(3)	89.90(16)
N(1)-C(1)	1.356(5)	O-N(1)-C(1)	119.0(3)
N(1)-C(5)	1.362(6)	O-N(1)-C(5)	121.0(3)
N(2)-C(6)	1.327(5)	C(1)-N(1)-C(5)	120.0(4)
N(2)-C(7)	1.472(5)	C(6)-N(2)-C(7)	123.6(4)
N(2)-C(8)	1.473(5)	C(6)-N(2)-C(8)	122.4(3)
N(3)-C(6)	1.324(5)	C(7)-N(2)-C(8)	113.7(3)
N(3)-C(9)	1.471(5)	C(6)-N(3)-C(9)	122.7(3)
N(3)-C(10)	1.473(5)	C(6)-N(3)-C(10)	123.5(3)
C(1)-C(2)	1.383(6)	C(9)-N(3)-C(10)	113.6(3)
C(2)-C(3)	1.378(6)	N(1)-C(1)-C(2)	120.4(4)
C(3)-C(4)	1.391(6)	N(1)-C(1)-S	112.1(3)
C(4)-C(5)	1.370(6)	C(2)-C(1)-S	127.5(3)
		C(3)-C(2)-C(1)	120.3(4)
C(1)-S-C(6)	98.33(18)	C(2)-C(3)-C(4)	118.4(4)
F(6)-P-F(5)	90.53(18)	C(5)-C(4)-C(3)	120.3(4)
F(6)-P-F(1)	179.01(18)	N(1)-C(5)-C(4)	120.6(4)
F(5)-P-F(1)	90.39(17)	N(3)-C(6)-N(2)	124.0(4)
F(6)-P-F(4)	89.31(16)	N(3)-C(6)-S	119.4(3)
F(5)-P-F(4)	90.89(16)	N(2)-C(6)-S	116.4(3)
F(1)-P-F(4)	90.30(16)		

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for $C_{10} H_{16} F_6 N_3 O P S$. 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}
S	28(1)	23(1)	34(1)	0(1)	6(1)	-1(1)
P	33(1)	21(1)	20(1)	0(1)	6(1)	-2(1)
F(1)	56(2)	53(2)	27(1)	13(1)	11(1)	-2(1)
F(2)	38(2)	38(2)	47(2)	-2(1)	3(1)	-10(1)
F(3)	55(2)	33(1)	46(2)	-14(1)	16(1)	2(1)
F(4)	37(1)	33(1)	45(2)	-3(1)	7(1)	-10(1)
F(5)	57(2)	32(1)	68(2)	-14(1)	25(2)	4(1)
F(6)	64(2)	65(2)	32(1)	24(1)	11(1)	3(2)
O	39(2)	28(2)	52(2)	-6(1)	-2(2)	-5(1)
N(1)	33(2)	24(2)	23(2)	1(1)	4(1)	-2(1)
N(2)	34(2)	26(2)	20(2)	-1(1)	6(1)	4(1)
N(3)	31(2)	23(2)	20(2)	0(1)	7(1)	-1(1)
C(1)	34(2)	22(2)	19(2)	1(2)	8(2)	2(2)
C(2)	29(2)	26(2)	22(2)	-2(2)	3(2)	-2(2)
C(3)	29(2)	35(2)	27(2)	4(2)	2(2)	-1(2)
C(4)	35(2)	34(2)	33(2)	8(2)	9(2)	11(2)
C(5)	47(3)	23(2)	28(2)	1(2)	10(2)	8(2)
C(6)	27(2)	21(2)	17(2)	0(1)	-1(2)	4(2)
C(7)	39(2)	47(3)	24(2)	-1(2)	12(2)	4(2)
C(8)	58(3)	23(2)	33(2)	-2(2)	10(2)	8(2)
C(9)	46(3)	36(2)	20(2)	-3(2)	11(2)	6(2)
C(10)	49(3)	30(2)	34(2)	1(2)	14(2)	-9(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $C_{10}H_{16}F_6N_3OPS$.

	x	y	z	U(eq)
H(2A)	-4009	6557	797	33
H(3A)	-6982	7004	206	39
H(4A)	-6763	7802	1244	42
H(5A)	-3636	8145	2682	40
H(7A)	1303	5739	-52	54
H(7B)	1943	6261	797	54
H(7C)	-307	6171	-516	54
H(7D)	656	6375	204	54
H(7E)	16	5853	-644	54
H(7F)	2267	5943	669	54
H(8A)	1052	5206	1509	60
H(8B)	-714	5223	2303	60
H(8C)	1562	5344	3498	60
H(8D)	215	5309	3365	60
H(8E)	1981	5292	2570	60
H(8F)	-295	5171	1375	60
H(9A)	-2401	6210	5767	51
H(9B)	-2162	6688	4765	51
H(9C)	-198	6416	6021	51
H(9D)	-773	6666	5268	51
H(9E)	-1012	6188	6270	51
H(9F)	-2976	6460	5015	51
H(10A)	-3486	5630	4275	57
H(10B)	-2122	5360	3360	57
H(10C)	-3955	5698	2247	57
H(10D)	-2889	5496	2313	57
H(10E)	-4253	5765	3228	57
H(10F)	-2421	5428	4341	57

Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Crystal data for $C_{10}H_{16}F_6N_3O_1P_1S_1$: $M_r = 371.28$, monoclinic, space group $P2_1/c$, a = 7.063(4), $b = 27.66(2)$, $c = 8.258(4)$ Å, $\beta = 110.45(4)°$, $V = 1512(2)$ Å^3, $Z = 4$, $D_c = 1.632$ g cm^{-3}, $\mu = 0.388$ mm^{-1}, $F(000) = 760$, $T = 150$ K, Refinement for data with $I > 2\sigma(I)$(3421 reflections) gave R1(F) = 0.0580 and wR2(F^2) = 0.1588.
(a) R1 is based on F, and wR2 is based upon F^2.
(b) Solved with *SHELXTL Version 5.1*, Bruker AXS, Inc., Madison, WI, 1997.

Intensity data was collected on a Syntex $P2_1$ diffractometer using Mo K_α radiation ($\lambda = 0.71073$ Å). The structure was solved using difference-Fourier syntheses (SHELXTL Version 5) [i] and were refined on $|F_o^2|$ using full-matrix least-squares . $C_{10}H_{16}F_6N_3O_1P_1S_1$: All hydrogen atoms (including the methyl hydrogens treated with a disorder model) were placed in calculated positions and refined using a riding model. All non-hydrogen atoms were refined anisotropically. Refinement of 2614 data against all 3421 unique data with no restraints yielded an R index, wR2 [ii], of 15.88% (R1 [iii] = 5.80% for $I > 2\sigma(I)$) with a goodness of fit [iv] of 1.089.

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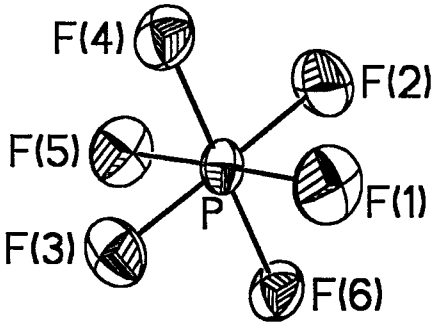
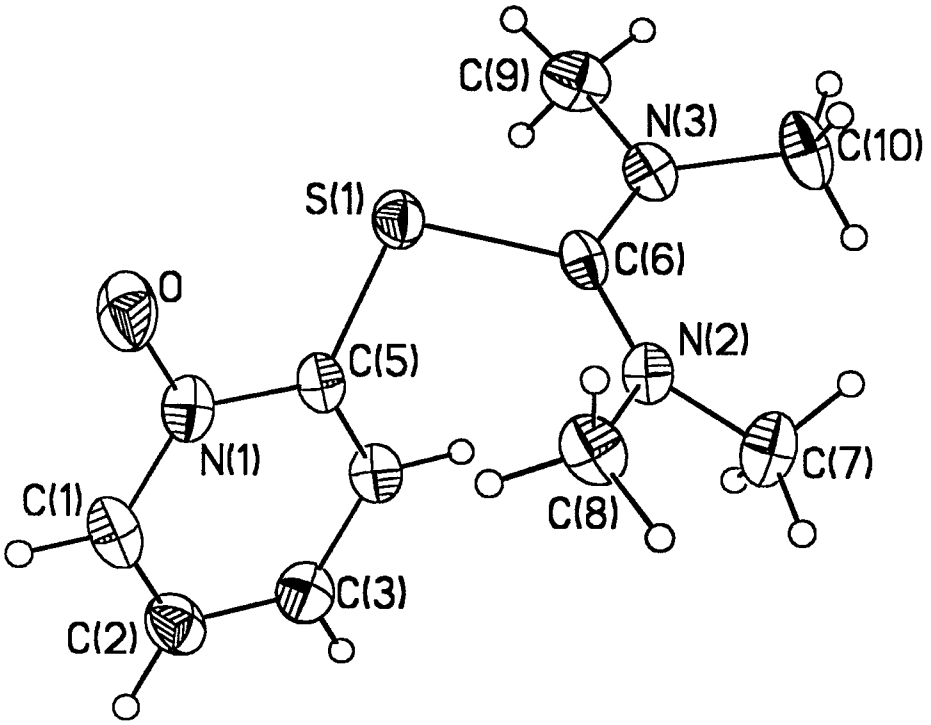
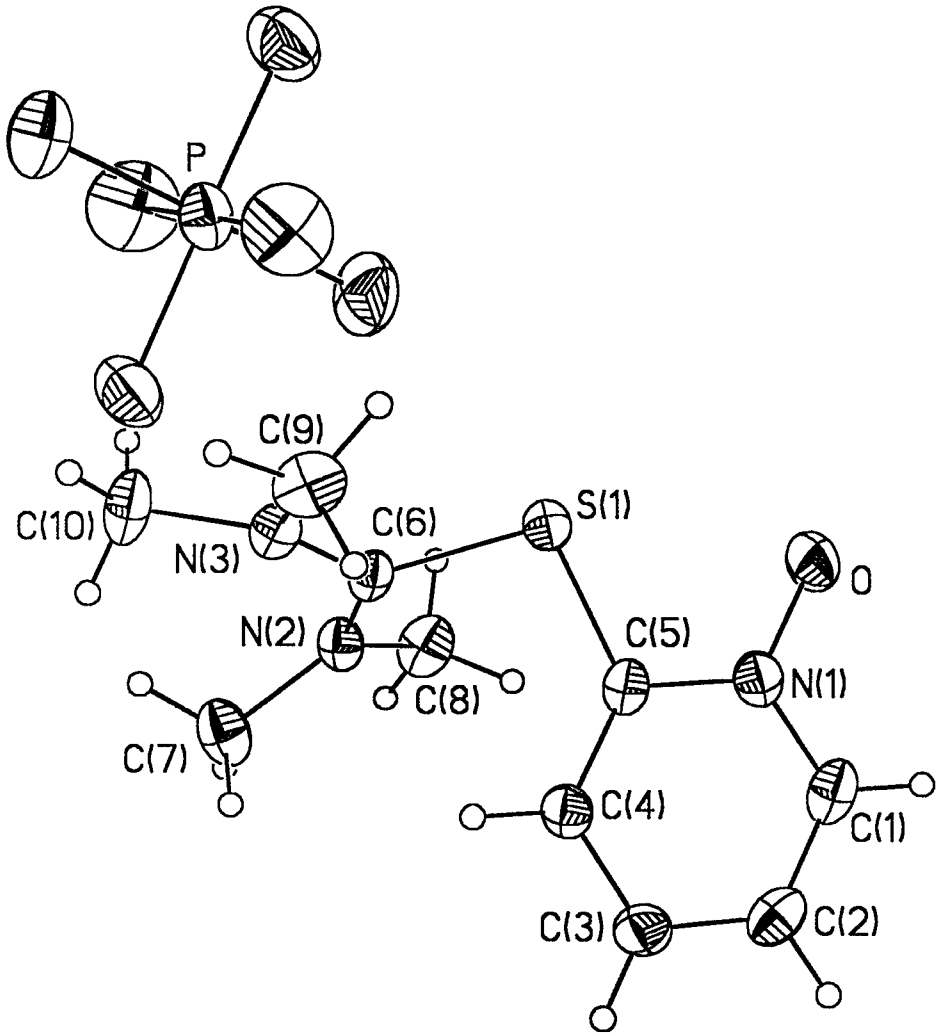
[i] *SHELXTL Version 5* Siemens Analytical X-Ray Instruments, Inc., Madison, WI, **September 1,1994**.
[ii] $wR2 = [\ \Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$ Weighted R-factors are based on F^2 and are statistically about twice as large as those based on F.
[iii] $R1 = \Sigma\,||F_o| - |F_c|\,| / \Sigma\,|F_o|$ Conventional R-factors are calculated using the observed criterion. This criterion is irrelevant to the choice of reflections used in the refinement.
[iv] $S = [\ \Sigma[w(F_o^2 - F_c^2)^2] / (n - p)]^{1/2}$ The goodness of fit is based on F^2 where: n = number of data and p = number of parameters refined.


F(4)
F(2)
F(5)
P
F(1)
F(3)
F(6)


C(9)
N(3)
C(10)
S(1)
C(6)
O
N(2)
C(5)
N(1)
C(7)
C(1)
C(8)
C(3)
C(2)


P
C(9)
C(10)
N(3)
C(6)
S(1)
O
N(2)
C(5)
N(1)
C(8)
C(7)
C(4)
C(1)
C(3)
C(2)

```
TITL     p21 in P2(1)/c
CELL 1
SFAC C  H  N  O  F  P  S
O     4    0.00000    0.00000    0.00000
P     6   -2.14885    8.11954    2.08240
S1    7   -1.99773    1.65070    0.78265
N1    3   -0.34011   -0.30857    1.22437
N2    3   -4.34796    0.76350    1.75377
N3    3   -3.94876    3.06503    1.90873
C1    1    0.28011   -1.32697    1.88937
H1A   2    0.94466   -1.84147    1.44629
C2    1   -0.04501   -1.61487    3.18776
H2A   2    0.37665   -2.34118    3.63177
C3    1   -0.99281   -0.84555    3.85987
H3A   2   -1.20123   -1.01649    4.77087
C4    1   -1.62113    0.17036    3.17014
H4A   2   -2.26875    0.70981    3.60792
C5    1   -1.31046    0.40644    1.84157
C6    1   -3.58093    1.83088    1.58617
C7    1   -5.35655    0.64640    2.82258
H7A   2   -5.80304   -0.22347    2.75640
H7B   2   -6.01907    1.36192    2.72632
H7C   2   -4.91762    0.72263    3.69546
C8    1   -4.18116   -0.46871    0.97121
H8A   2   -4.84865   -1.12821    1.25425
H8B   2   -3.28226   -0.82993    1.11825
H8C   2   -4.30190   -0.26982    0.01923
C9    1   -2.99634    4.16946    2.09979
H9A   2   -3.48567    4.98628    2.33198
H9B   2   -2.49357    4.31432    1.27119
H9C   2   -2.37545    3.94387    2.82358
C10   1   -5.36191    3.47812    1.97719
H10A  2   -5.41412    4.42318    2.23121
H10B  2   -5.82934    2.93406    2.64503
H10C  2   -5.78220    3.35178    1.10093
F1    5   -3.44994    7.46129    1.44487
F2    5   -2.93858    9.48047    2.31501
F3    5   -1.33221    6.75790    1.84711
F4    5   -1.68468    8.64521    0.64124
F5    5   -0.83433    8.75770    2.72995
F6    5   -2.60683    7.57608    3.52087

END
FMOL
MPLN
UNDO

LINK O    N1     1
LINK N1   C1     1
LINK C1   H1A    1
LINK C1   C2     1
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LINK C2    C3      1
LINK C3    H3A     1
LINK C3    C4      1
LINK C4    H4A     1
LINK S1    C5      1
LINK N1    C5      1
LINK C4    C5      1
LINK S1    C6      1
LINK N2    C6      1
LINK N3    C6      1
LINK N2    C7      1
LINK C7    H7A     1
LINK C7    H7B     1
LINK C7    H7C     1
LINK N2    C8      1
LINK C8    H8A     1
LINK C8    H8B     1
LINK C8    H8C     1
LINK N3    C9      1
LINK C9    H9A     1
LINK C9    H9B     1
LINK C9    H9C     1
LINK N3    C10     1
LINK C10   H10A    1
LINK C10   H10B    1
LINK C10   H10C    1
LINK P     F1      1
LINK P     F2      1
LINK P     F3      1
LINK P     F4      1
LINK P     F5      1
LINK P     F6      1

END
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