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Design, Synthesis and Evaluation of A Novel Glutamine Derivative (*2S,4R*)-2-amino-4-cyano-4-[^{18}F]fluoro-butanoic acid

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1. In vitro stability (*2S,4R*)4-[^{18}F]FGln and [^{18}F]1

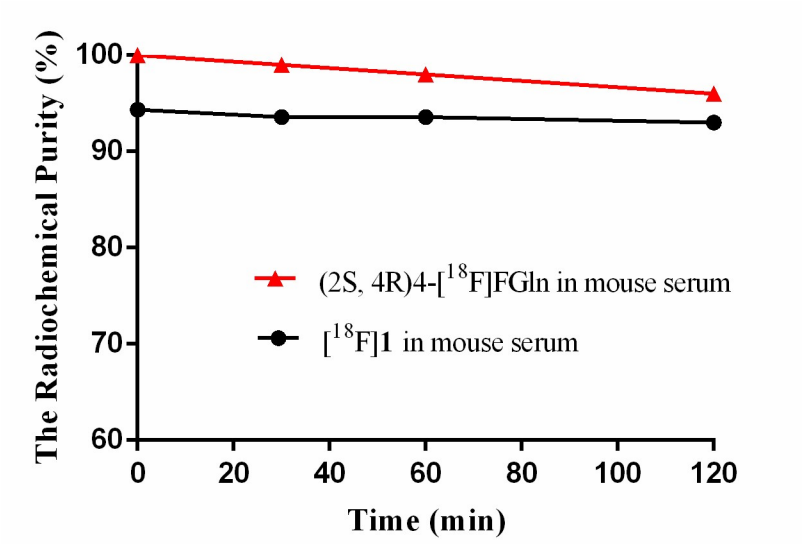
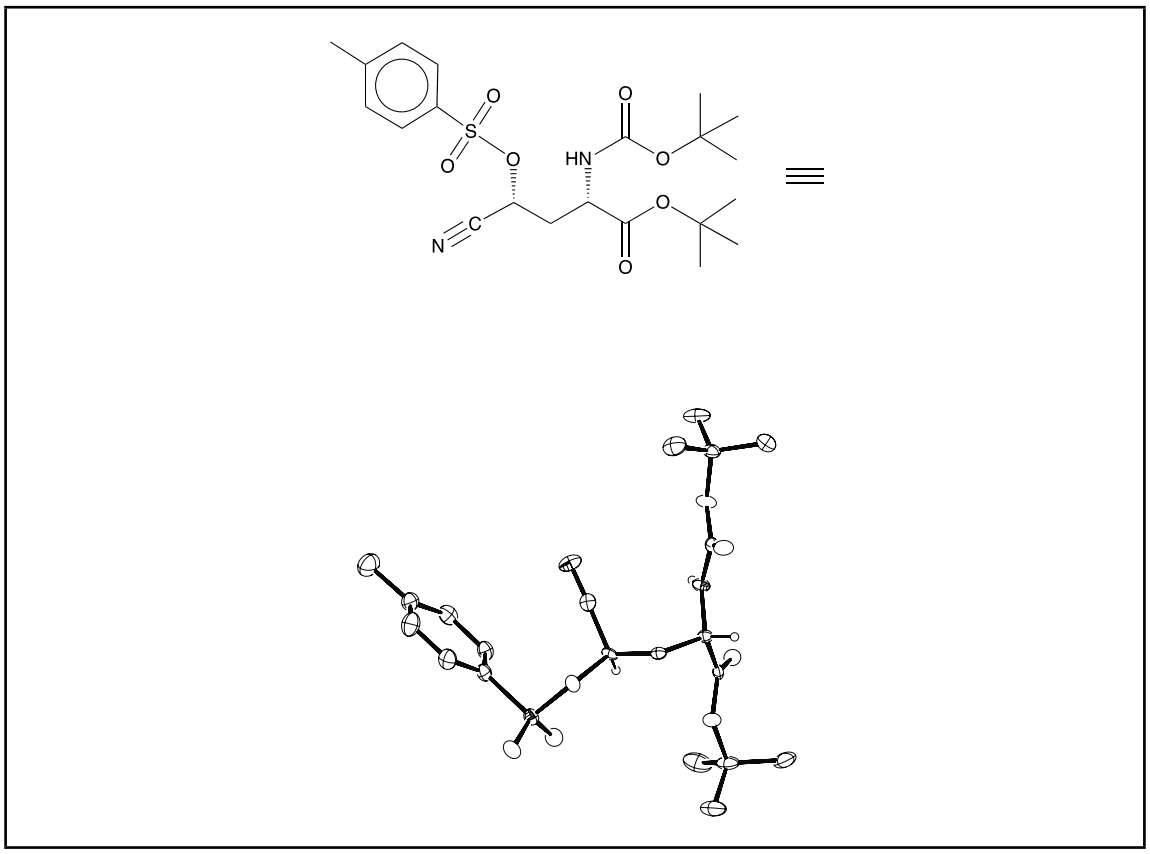


Figure S1. In vitro stability of (*2S,4R*)4-[^{18}F]FGln and **[^{18}F]1** in mouse serum buffers.

2. X-ray Structure Determination of radiolabeling precursor 4



Compound **4**, $C_{21}H_{30}N_2O_7S$, crystallizes in the orthorhombic space group $P2_12_12_1$ (systematic absences h00: h=odd, 0k0: k=odd, and 00l: l=odd) with a=5.9695(2)Å, b=10.9627(3)Å, c=36.0572(11)Å, V=2359.65(12)Å^3, Z=4, and d_{calc}=1.279 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 2348 frames were collected with a crystal to detector distance of 37.6 mm, rotation widths of 0.5° and exposures of 20 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	346.20	14.21	32.61	739
ω	-23.00	320.59	91.38	-46.47	265
ω	14.50	42.88	65.26	-33.72	162
ω	24.50	278.77	12.44	23.24	271
ϕ	-23.00	336.61	24.44	52.47	739
ϕ	24.50	68.72	341.49	-42.87	60

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 33855 reflections were measured over the ranges $1.13 \leq \theta \leq 25.42°$, $-7 \leq h \leq 7$, $-13 \leq k \leq 13$, $-39 \leq l \leq 43$ yielding 4341 unique reflections (Rint = 0.0455). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6775, 0.7452).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0314P)^2 + 1.2540P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0391 and wR2=0.0868 for 4016 observed reflections for which $F > 4\sigma(F)$ and R1=0.0445 and wR2=0.0890 and GOF =1.185 for all 4341 unique, non-zero reflections and 288 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.229 and -0.289 e/Å^3.

Table S1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables S2. and S3. Anisotropic thermal parameters are in Table S4. Tables S5. and S6. list bond distances and bond angles. Figure S1. is an ORTEP[vii] representation of the molecule with 50% probability thermal ellipsoids displayed.

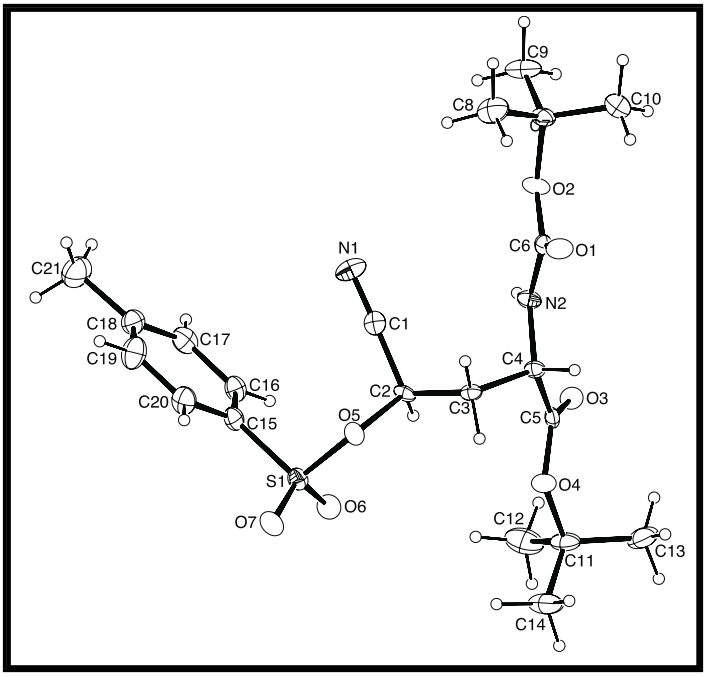


Figure S1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table S1. Summary of Structure Determination of Compound 4

Empirical formula	$C_{21}H_{30}N_2O_7S$
Formula weight	454.53
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	$P2_12_12_1$
Cell constants:	
a	5.9695(2) Å
b	10.9627(3) Å
c	36.0572(11) Å
Volume	2359.65(12) $Å^3$
Z	4
Density (calculated)	1.279 Mg/m^3
Absorption coefficient	0.180 mm^{-1}
F(000)	968
Crystal size	0.50 x 0.10 x 0.02 mm^3
Theta range for data collection	1.13 to 25.42°
Index ranges	$-7 \leq h \leq 7$, $-13 \leq k \leq 13$, $-39 \leq l \leq 43$
Reflections collected	33855
Independent reflections	4341 [R(int) = 0.0455]
Completeness to theta = 25.42°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6775
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4341 / 0 / 288
Goodness-of-fit on F^2	1.185
Final R indices [I>2sigma(I)]	R1 = 0.0391, wR2 = 0.0868
R indices (all data)	R1 = 0.0445, wR2 = 0.0890
Absolute structure parameter	0.07(8)
Largest diff. peak and hole	0.229 and -0.289 $e.Å^{-3}$

Table S2. Refined Positional Parameters for Compound 4

Atom	x	y	z	U_{eq}, Å^2
C1	0.4172(4)	0.2571(2)	0.37396(6)	0.0176(5)
C2	0.3140(4)	0.3805(2)	0.37229(6)	0.0142(5)
C3	0.3161(4)	0.4349(2)	0.33351(7)	0.0140(5)
C4	0.5462(4)	0.4859(2)	0.32238(6)	0.0142(5)
C5	0.6241(4)	0.5909(2)	0.34658(6)	0.0143(5)
C6	0.7432(4)	0.3203(2)	0.29170(7)	0.0155(5)
C7	0.9762(4)	0.1491(2)	0.27034(6)	0.0192(5)
C8	0.7779(5)	0.0657(3)	0.26521(9)	0.0307(7)
C9	1.1640(5)	0.0829(3)	0.28987(8)	0.0305(7)
C10	1.0550(6)	0.2051(3)	0.23445(8)	0.0388(8)
C11	0.4827(5)	0.7820(2)	0.37319(7)	0.0247(6)
C12	0.5746(5)	0.7606(3)	0.41155(8)	0.0385(8)
C13	0.6264(5)	0.8622(2)	0.34895(9)	0.0371(8)
C14	0.2440(5)	0.8298(3)	0.37509(9)	0.0346(7)
C15	0.0723(4)	0.2417(2)	0.44504(7)	0.0186(5)
C16	0.2767(4)	0.2259(2)	0.46291(7)	0.0200(5)
C17	0.3242(4)	0.1121(3)	0.47786(7)	0.0236(6)
C18	0.1747(5)	0.0164(2)	0.47600(7)	0.0251(6)
C19	-0.0281(5)	0.0360(3)	0.45750(8)	0.0303(7)
C20	-0.0796(4)	0.1472(2)	0.44183(7)	0.0247(6)
C21	0.2234(5)	-0.1048(3)	0.49386(8)	0.0381(7)
N1	0.4913(4)	0.16157(19)	0.37542(6)	0.0281(5)
N2	0.7206(3)	0.39461(18)	0.32135(5)	0.0161(4)
O1	0.6224(3)	0.32233(15)	0.26491(5)	0.0208(4)
O2	0.9207(3)	0.24629(15)	0.29720(5)	0.0188(4)
O3	0.8144(3)	0.60465(15)	0.35662(5)	0.0190(4)
O4	0.4510(3)	0.66398(15)	0.35400(5)	0.0184(4)
O5	0.0782(2)	0.36782(15)	0.38274(4)	0.0165(4)
O6	0.1446(3)	0.47560(16)	0.44149(5)	0.0231(4)
O7	-0.2297(3)	0.39761(16)	0.42310(5)	0.0235(4)
S1	0.00798(10)	0.38350(5)	0.425200(15)	0.01749(14)

$U_{eq}={}^1/_3[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table S3. Positional Parameters for Hydrogens in Compound 4

Atom	x	y	z	U_{iso}, Å^2
H2	0.3903	0.4354	0.3896	0.019
H3a	0.2062	0.5000	0.3323	0.019
H3b	0.2723	0.3726	0.3158	0.019
H4	0.5311	0.5179	0.2971	0.019
H8a	0.7248	0.0392	0.2890	0.046
H8b	0.8220	-0.0039	0.2508	0.046
H8c	0.6606	0.1087	0.2525	0.046
H9a	1.2885	0.1373	0.2931	0.046
H9b	1.2100	0.0142	0.2752	0.046
H9c	1.1129	0.0551	0.3137	0.046
H10a	0.9355	0.2517	0.2236	0.058
H10b	1.0994	0.1417	0.2176	0.058
H10c	1.1803	0.2577	0.2392	0.058
H12a	0.4866	0.6995	0.4238	0.058
H12b	0.5686	0.8352	0.4255	0.058
H12c	0.7271	0.7335	0.4098	0.058
H13a	0.7751	0.8292	0.3477	0.056
H13b	0.6320	0.9429	0.3593	0.056
H13c	0.5635	0.8657	0.3245	0.056
H14a	0.1834	0.8352	0.3505	0.052
H14b	0.2437	0.9092	0.3863	0.052
H14c	0.1542	0.7752	0.3896	0.052
H16	0.3787	0.2897	0.4648	0.027
H17	0.4613	0.1001	0.4895	0.031
H19	-0.1308	-0.0275	0.4557	0.040
H20	-0.2143	0.1584	0.4293	0.033
H21a	0.3819	-0.1130	0.4976	0.057
H21b	0.1715	-0.1693	0.4780	0.057
H21c	0.1480	-0.1095	0.5173	0.057
H2a	0.8110	0.3875	0.3398	0.021

Table S4. Refined Thermal Parameters (U's) for Compound 4

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0166(12)	0.0197(13)	0.0164(12)	-0.0014(10)	-0.0030(10)	-0.0042(11)
C2	0.0063(10)	0.0185(12)	0.0177(12)	-0.0030(10)	0.0014(9)	0.0024(11)
C3	0.0112(12)	0.0126(11)	0.0181(13)	-0.0027(10)	-0.0012(10)	0.0016(10)
C4	0.0109(12)	0.0147(11)	0.0169(12)	-0.0007(9)	-0.0007(9)	0.0022(10)
C5	0.0118(12)	0.0163(13)	0.0147(12)	0.0041(9)	0.0032(9)	-0.0022(10)
C6	0.0159(13)	0.0133(11)	0.0173(13)	0.0002(10)	0.0035(10)	-0.0032(10)
C7	0.0188(13)	0.0165(12)	0.0222(13)	-0.0077(9)	0.0034(11)	-0.0005(11)
C8	0.0238(15)	0.0234(14)	0.0451(18)	-0.0071(13)	-0.0045(13)	-0.0029(12)
C9	0.0243(15)	0.0249(15)	0.0424(18)	-0.0151(13)	-0.0055(12)	0.0089(12)
C10	0.051(2)	0.0292(15)	0.0364(17)	0.0001(13)	0.0233(15)	0.0062(14)
C11	0.0186(13)	0.0170(12)	0.0384(15)	-0.0134(11)	0.0005(12)	0.0003(12)
C12	0.0318(17)	0.0455(18)	0.0381(17)	-0.0202(15)	-0.0018(13)	0.0041(15)
C13	0.0280(15)	0.0144(14)	0.069(2)	-0.0017(13)	0.0103(15)	0.0018(12)
C14	0.0202(15)	0.0252(15)	0.058(2)	-0.0168(14)	0.0056(14)	0.0012(12)
C15	0.0194(13)	0.0230(13)	0.0134(12)	0.0022(10)	0.0011(10)	-0.0004(11)
C16	0.0183(13)	0.0259(13)	0.0159(13)	0.0014(11)	-0.0006(10)	-0.0023(11)
C17	0.0226(13)	0.0334(15)	0.0147(13)	0.0003(12)	-0.0029(10)	0.0021(13)
C18	0.0347(16)	0.0239(14)	0.0166(14)	0.0022(11)	0.0022(11)	0.0031(13)
C19	0.0303(16)	0.0285(15)	0.0323(15)	0.0033(12)	-0.0044(13)	-0.0105(13)
C20	0.0178(13)	0.0276(15)	0.0288(14)	0.0018(11)	-0.0022(11)	-0.0044(11)
C21	0.0482(18)	0.0333(16)	0.0329(16)	0.0077(14)	-0.0028(14)	0.0060(16)
N1	0.0309(13)	0.0185(11)	0.0350(13)	0.0000(9)	-0.0062(11)	0.0078(11)
N2	0.0120(9)	0.0188(11)	0.0173(10)	-0.0035(9)	-0.0015(8)	0.0032(9)
O1	0.0207(9)	0.0208(9)	0.0208(9)	-0.0040(7)	-0.0039(8)	0.0049(8)
O2	0.0119(8)	0.0216(9)	0.0230(9)	-0.0094(7)	-0.0011(7)	0.0033(7)
O3	0.0106(8)	0.0178(9)	0.0287(9)	-0.0011(8)	-0.0010(7)	-0.0029(7)
O4	0.0118(9)	0.0159(8)	0.0275(9)	-0.0058(7)	-0.0002(7)	-0.0011(7)
O5	0.0094(8)	0.0221(9)	0.0179(8)	0.0023(7)	-0.0006(6)	-0.0015(7)
O6	0.0260(10)	0.0220(9)	0.0213(9)	-0.0041(8)	0.0019(8)	-0.0041(8)
O7	0.0139(8)	0.0314(10)	0.0253(10)	0.0048(8)	0.0036(7)	0.0045(8)
S1	0.0144(3)	0.0205(3)	0.0176(3)	0.0009(2)	0.0013(2)	-0.0007(3)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

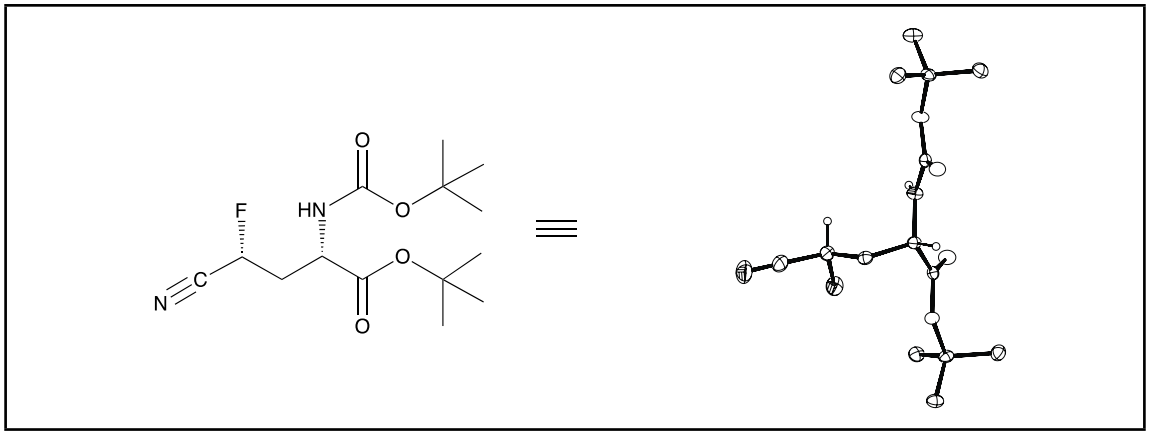
Table S5. Bond Distances in Compound 4, Å

C1-N1	1.138(3)	C1-C2	1.487(3)	C2-O5	1.464(3)
C2-C3	1.521(3)	C3-C4	1.536(3)	C4-N2	1.445(3)
C4-C5	1.517(3)	C5-O3	1.202(3)	C5-O4	1.334(3)
C6-O1	1.206(3)	C6-O2	1.349(3)	C6-N2	1.351(3)
C7-O2	1.478(3)	C7-C8	1.507(4)	C7-C10	1.508(4)
C7-C9	1.510(4)	C11-O4	1.479(3)	C11-C12	1.506(4)
C11-C13	1.508(4)	C11-C14	1.520(4)	C15-C20	1.382(3)
C15-C16	1.391(3)	C15-S1	1.754(3)	C16-C17	1.388(4)
C17-C18	1.380(4)	C18-C19	1.399(4)	C18-C21	1.505(4)
C19-C20	1.378(4)	O5-S1	1.5965(16)	O6-S1	1.4248(18)
O7-S1	1.4291(17)				

Table S6. Bond Angles in Compound 4, °

N1-C1-C2	178.4(3)	O5-C2-C1	107.57(19)	O5-C2-C3	106.37(17)
C1-C2-C3	113.01(19)	C2-C3-C4	112.97(19)	N2-C4-C5	108.63(18)
N2-C4-C3	113.52(18)	C5-C4-C3	113.57(19)	O3-C5-O4	126.6(2)
O3-C5-C4	123.9(2)	O4-C5-C4	109.49(19)	O1-C6-O2	126.8(2)
O1-C6-N2	124.3(2)	O2-C6-N2	108.9(2)	O2-C7-C8	110.0(2)
O2-C7-C10	109.78(19)	C8-C7-C10	112.8(2)	O2-C7-C9	101.96(19)
C8-C7-C9	110.4(2)	C10-C7-C9	111.4(2)	O4-C11-C12	109.9(2)
O4-C11-C13	108.2(2)	C12-C11-C13	114.6(3)	O4-C11-C14	101.7(2)
C12-C11-C14	110.7(2)	C13-C11-C14	111.0(2)	C20-C15-C16	121.4(2)
C20-C15-S1	119.13(19)	C16-C15-S1	119.46(19)	C17-C16-C15	118.1(2)
C18-C17-C16	122.2(2)	C17-C18-C19	117.8(2)	C17-C18-C21	121.7(3)
C19-C18-C21	120.5(3)	C20-C19-C18	121.7(3)	C19-C20-C15	118.8(2)
C6-N2-C4	120.7(2)	C6-O2-C7	120.88(18)	C5-O4-C11	121.34(18)
C2-O5-S1	119.29(14)	O6-S1-O7	120.90(11)	O6-S1-O5	108.74(10)
O7-S1-O5	102.80(10)	O6-S1-C15	109.54(11)	O7-S1-C15	109.57(11)
O5-S1-C15	103.78(10)				

3. X-ray Structure Determination of Intermediate 7



Compound **7**, $C_{14}H_{23}N_2O_4F$, crystallizes in the orthorhombic space group $P2_12_12_1$ (systematic absences h00: h=odd, 0k0: k=odd, and 00l: l=odd) with a=5.33170(10)Å, b=10.0576(3)Å, c=30.0104(8)Å, V=1609.28(7)Å^3, Z=4, and d_{calc}=1.248 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 100(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 4434 frames were collected with a crystal to detector distance of 44.8 mm, rotation widths of 0.5° and exposures of 15 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-28.00	340.03	328.61	-63.64	739
ϕ	14.50	101.06	337.92	-48.25	739
ω	-28.00	220.69	190.55	45.39	245
ω	-28.00	213.74	117.50	57.63	254
ϕ	-28.00	327.36	13.34	30.75	415
ϕ	-23.00	328.91	44.82	-70.01	577
ϕ	-18.00	99.81	333.80	-58.65	726
ϕ	27.00	43.00	330.80	-60.33	739

Rotation frames were integrated using SAINT[viii], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ix] program package for further processing and structure solution. A total of 39657 reflections were measured over the ranges $1.36 \leq \theta \leq 25.37°$, $-6 \leq h \leq 6$, $-12 \leq k \leq 12$, $-36 \leq l \leq 35$ yielding 2968 unique reflections (Rint

= 0.0293). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[x] (minimum and maximum transmission 0.7064, 0.7452).

The structure was solved by direct methods (SHELXS-97[xi]). Refinement was by full-matrix least squares based on F^2 using SHELXL-97.[xii] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0288P)^2 + 0.3182P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to R1=0.0252 and wR2=0.0595 for 2797 observed reflections for which $F > 4\sigma(F)$ and R1=0.0280 and wR2=0.0607 and GOF =1.093 for all 2968 unique, non-zero reflections and 197 variables.[xiii] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.158 and -0.171 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[xiv] representation of the molecule with 50% probability thermal ellipsoids displayed.

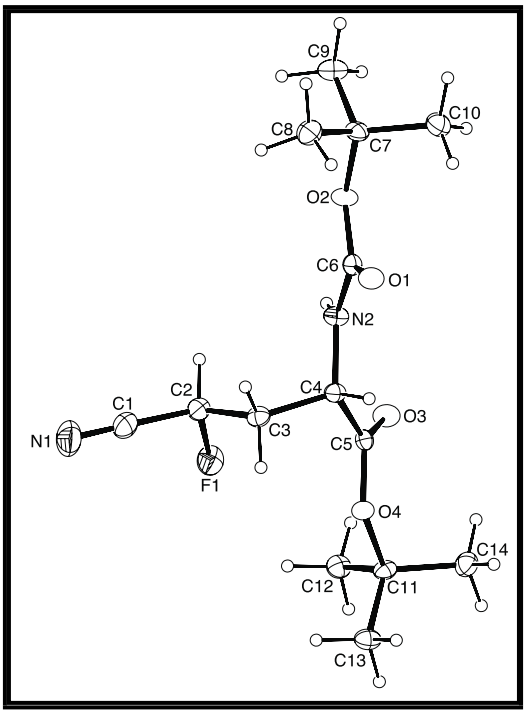


Figure 1. ORTEP drawing of the title compound with 50% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 7

Empirical formula	$C_{14}H_{23}N_2O_4F$
Formula weight	302.34
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	$P2_12_12_1$
Cell constants:	
a	5.33170(10) Å
b	10.0576(3) Å
c	30.0104(8) Å
Volume	1609.28(7) $Å^3$
Z	4
Density (calculated)	1.248 Mg/m^3
Absorption coefficient	0.098 mm^{-1}
F(000)	648
Crystal size	0.42 x 0.12 x 0.04 mm^3
Theta range for data collection	1.36 to 25.37°
Index ranges	$-6 \leq h \leq 6$, $-12 \leq k \leq 12$, $-36 \leq l \leq 35$
Reflections collected	39657
Independent reflections	2968 [R(int) = 0.0293]
Completeness to theta = 25.37°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.7064
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2968 / 0 / 197
Goodness-of-fit on F^2	1.093
Final R indices [I>2sigma(I)]	R1 = 0.0252, wR2 = 0.0595
R indices (all data)	R1 = 0.0280, wR2 = 0.0607
Absolute structure parameter	0.1(6)
Largest diff. peak and hole	0.158 and -0.171 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 7

Atom	x	y	z	U_{eq}, Å^2
C1	0.8389(3)	0.17852(14)	0.03725(5)	0.0253(3)
C2	0.8234(3)	0.25457(13)	0.07943(4)	0.0189(3)
C3	0.5620(2)	0.24979(12)	0.09902(4)	0.0161(3)
C4	0.5289(2)	0.33879(12)	0.14036(4)	0.0147(3)
C5	0.6919(2)	0.29757(12)	0.17968(4)	0.0138(3)
C6	0.3740(2)	0.55286(12)	0.11597(4)	0.0153(3)
C7	0.2806(2)	0.77500(13)	0.08729(4)	0.0167(3)
C8	0.1391(3)	0.72289(14)	0.04704(4)	0.0228(3)
C9	0.4565(3)	0.88653(13)	0.07365(5)	0.0239(3)
C10	0.1086(3)	0.82034(14)	0.12436(4)	0.0223(3)
C11	0.7944(2)	0.09957(13)	0.22433(4)	0.0152(3)
C12	1.0732(2)	0.10867(14)	0.21520(5)	0.0186(3)
C13	0.7059(3)	-0.04328(13)	0.21971(5)	0.0193(3)
C14	0.7266(3)	0.15624(14)	0.26953(4)	0.0207(3)
N1	0.8506(3)	0.11896(14)	0.00507(4)	0.0414(4)
N2	0.5687(2)	0.47709(10)	0.12976(3)	0.0159(2)
O1	0.15768(17)	0.51509(9)	0.11433(3)	0.0184(2)
O2	0.45668(16)	0.67453(8)	0.10467(3)	0.01736(19)
O3	0.83022(18)	0.37186(9)	0.19927(3)	0.0200(2)
O4	0.65151(16)	0.16966(8)	0.18879(3)	0.01432(19)
F1	0.99360(14)	0.19537(8)	0.10882(3)	0.02547(19)

$U_{eq}=^1/_3[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 7

Atom	x	y	z	U_{iso}, Å^2
H2	0.8719	0.3472	0.0741	0.025
H3a	0.5233	0.1587	0.1071	0.021
H3b	0.4427	0.2772	0.0764	0.021
H4	0.3539	0.3299	0.1499	0.020
H8a	0.2519	0.6740	0.0282	0.034
H8b	0.0700	0.7962	0.0307	0.034
H8c	0.0060	0.6655	0.0568	0.034
H9a	0.5520	0.9149	0.0990	0.036
H9b	0.3603	0.9600	0.0624	0.036
H9c	0.5682	0.8553	0.0509	0.036
H10a	-0.0029	0.7492	0.1323	0.033
H10b	0.0126	0.8955	0.1145	0.033

H10c	0.2069	0.8451	0.1499	0.033
H12a	1.1068	0.0793	0.1854	0.028
H12b	1.1623	0.0533	0.2359	0.028
H12c	1.1273	0.1992	0.2185	0.028
H13a	0.5283	-0.0474	0.2248	0.029
H13b	0.7907	-0.0978	0.2412	0.029
H13c	0.7429	-0.0749	0.1903	0.029
H14a	0.7819	0.2469	0.2713	0.031
H14b	0.8068	0.1050	0.2925	0.031
H14c	0.5480	0.1527	0.2735	0.031
H2a	0.7158	0.5113	0.1322	0.021

Table 4. Refined Thermal Parameters (U's) for Compound 7

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0280(7)	0.0239(7)	0.0241(7)	0.0019(6)	0.0073(6)	-0.0045(7)
C2	0.0211(7)	0.0181(6)	0.0176(6)	0.0006(5)	0.0007(6)	-0.0007(6)
C3	0.0169(6)	0.0159(6)	0.0155(6)	0.0014(5)	-0.0032(5)	-0.0008(5)
C4	0.0141(6)	0.0131(6)	0.0169(6)	0.0016(5)	0.0001(5)	0.0005(5)
C5	0.0125(6)	0.0145(6)	0.0146(6)	-0.0007(5)	0.0026(5)	0.0019(5)
C6	0.0182(6)	0.0151(6)	0.0126(6)	-0.0012(5)	0.0004(5)	0.0009(5)
C7	0.0173(6)	0.0147(6)	0.0182(6)	0.0016(5)	-0.0031(5)	0.0043(5)
C8	0.0263(7)	0.0240(7)	0.0181(7)	0.0018(5)	-0.0030(6)	0.0016(6)
C9	0.0248(7)	0.0176(7)	0.0293(7)	0.0052(6)	-0.0029(6)	0.0003(6)
C10	0.0251(7)	0.0199(7)	0.0219(7)	-0.0003(6)	-0.0002(6)	0.0059(6)
C11	0.0125(6)	0.0163(7)	0.0167(6)	0.0045(5)	-0.0025(5)	0.0026(5)
C12	0.0147(7)	0.0199(7)	0.0212(6)	0.0013(5)	-0.0011(5)	0.0021(6)
C13	0.0170(6)	0.0166(7)	0.0244(7)	0.0035(5)	-0.0019(6)	0.0020(6)
C14	0.0217(7)	0.0238(7)	0.0167(6)	0.0020(5)	0.0003(5)	0.0025(6)
N1	0.0576(9)	0.0387(8)	0.0277(7)	-0.0083(6)	0.0139(7)	-0.0073(8)
N2	0.0132(5)	0.0136(5)	0.0210(5)	0.0014(4)	-0.0027(5)	-0.0009(4)
O1	0.0147(4)	0.0170(4)	0.0237(5)	0.0025(4)	-0.0006(4)	0.0001(4)
O2	0.0152(4)	0.0124(4)	0.0244(5)	0.0039(4)	-0.0025(4)	0.0013(4)
O3	0.0230(5)	0.0164(4)	0.0205(5)	-0.0004(4)	-0.0062(4)	-0.0039(4)
O4	0.0133(4)	0.0136(4)	0.0160(4)	0.0020(3)	-0.0021(3)	0.0001(4)
F1	0.0176(4)	0.0333(5)	0.0255(4)	0.0014(4)	0.0001(3)	0.0039(3)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 7, Å

C1-N1	1.1381(19)	C1-C2	1.4812(19)	C2-F1	1.3985(15)
C2-C3	1.5135(18)	C3-C4	1.5398(17)	C4-N2	1.4425(16)
C4-C5	1.5229(17)	C5-O3	1.2034(15)	C5-O4	1.3327(15)
C6-O1	1.2152(16)	C6-O2	1.3442(15)	C6-N2	1.3526(16)
C7-O2	1.4746(15)	C7-C10	1.5122(18)	C7-C8	1.5175(18)
C7-C9	1.5182(19)	C11-O4	1.4881(14)	C11-C12	1.5144(18)
C11-C14	1.5154(18)	C11-C13	1.5185(18)		

Table 6. Bond Angles in Compound 7, °

N1-C1-C2	179.33(15)	F1-C2-C1	106.44(11)	F1-C2-C3	109.81(10)
C1-C2-C3	111.52(11)	C2-C3-C4	113.58(10)	N2-C4-C5	110.46(10)
N2-C4-C3	111.48(10)	C5-C4-C3	113.61(10)	O3-C5-O4	126.72(11)
O3-C5-C4	124.01(11)	O4-C5-C4	109.26(10)	O1-C6-O2	125.86(12)
O1-C6-N2	124.37(12)	O2-C6-N2	109.78(10)	O2-C7-C10	109.42(10)
O2-C7-C8	111.18(10)	C10-C7-C8	112.84(11)	O2-C7-C9	102.04(10)
C10-C7-C9	110.49(11)	C8-C7-C9	110.34(11)	O4-C11-C12	110.14(10)
O4-C11-C14	109.95(10)	C12-C11-C14	111.93(11)	O4-C11-C13	102.94(10)
C12-C11-C13	110.22(11)	C14-C11-C13	111.31(11)	C6-N2-C4	119.87(11)
C6-O2-C7	120.28(10)	C5-O4-C11	121.45(9)		

4. Reference

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.
[viii]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ix]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

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[xiii]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

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