

9050: 2nd attempt at N-terminus of Paxillin

(see p. 3-54 for full sequence)

part 1 (on the synthesizer):

$\text{NH}_2\text{-ISKRPVFLSEETPYSYPTG-O}$ (TGT resin)

part 2 (by hand b/c the His(Trt)/Ile coupling seemed to be the most problematic spot on the first synthesis attempt)

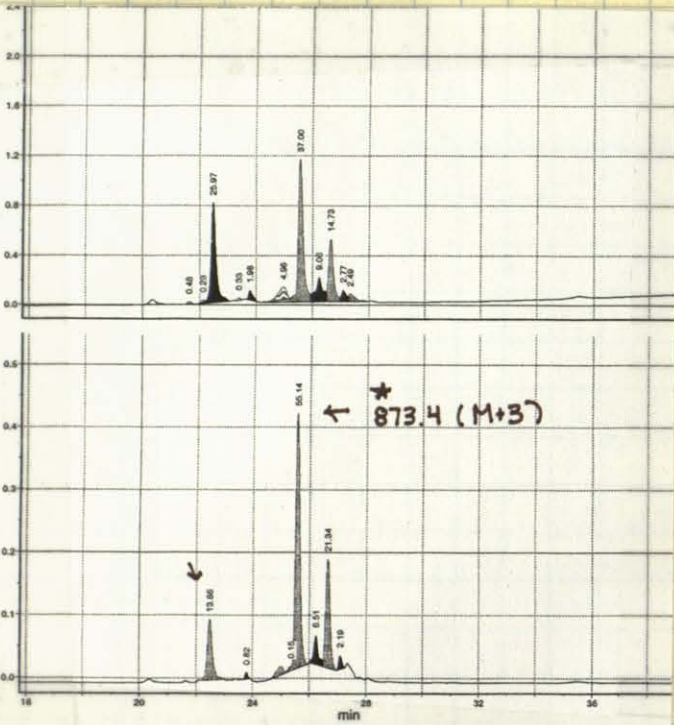
$\text{Fmoc-SHISKRPVFLSEETPYSYPTG-O}$

part 3 (new run on the synthesizer)

$\text{NH}_2\text{-DDL DALLADLESTSHISKRPVFLSEETPYSYPTG-O}$

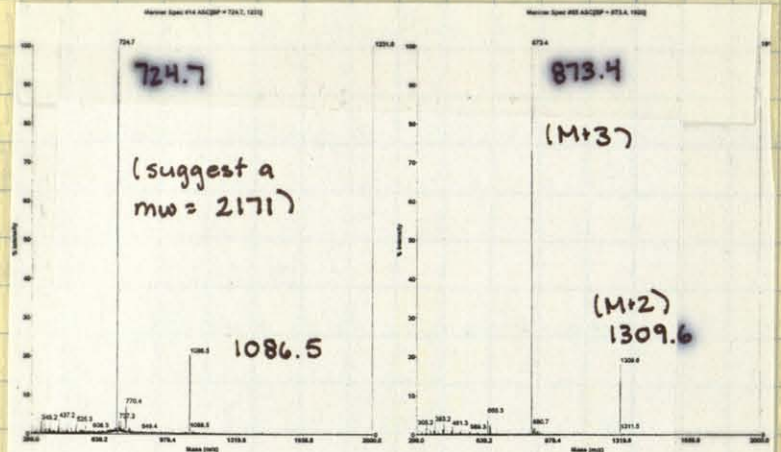
part 4 (by hand)

addition of final residue in 1-36 PAX (a Met)
and addition of a His4 purification tag



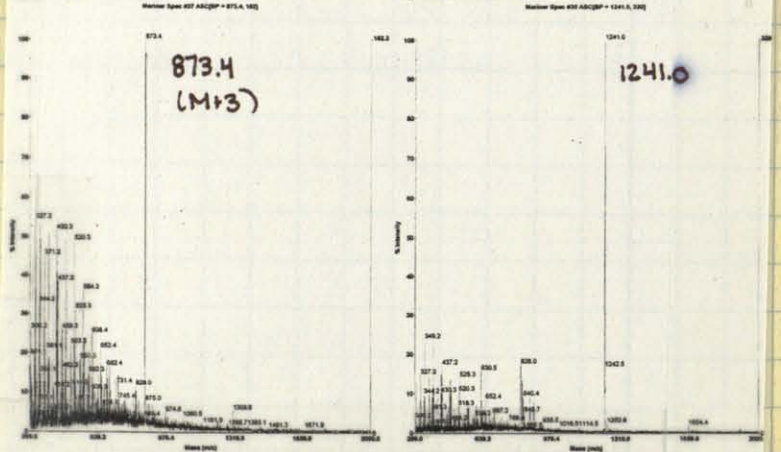
2/03/03 Fmoc-SHI...G-COOH

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Mariner Mass Spectrum
D:_V020403_FmocSHI_G_22p4001.dat
Acquired: Feb 04 08:55:00 2003

Mariner Mass Spectrum
D:_V020403_FmocSHI_G_25p3001.dat
Acquired: Feb 04 08:56:00 2003



Mariner Mass Spectrum
D:_V020403_FmocSHI_G_25p3001.dat
Acquired: Feb 04 09:00:00 2003

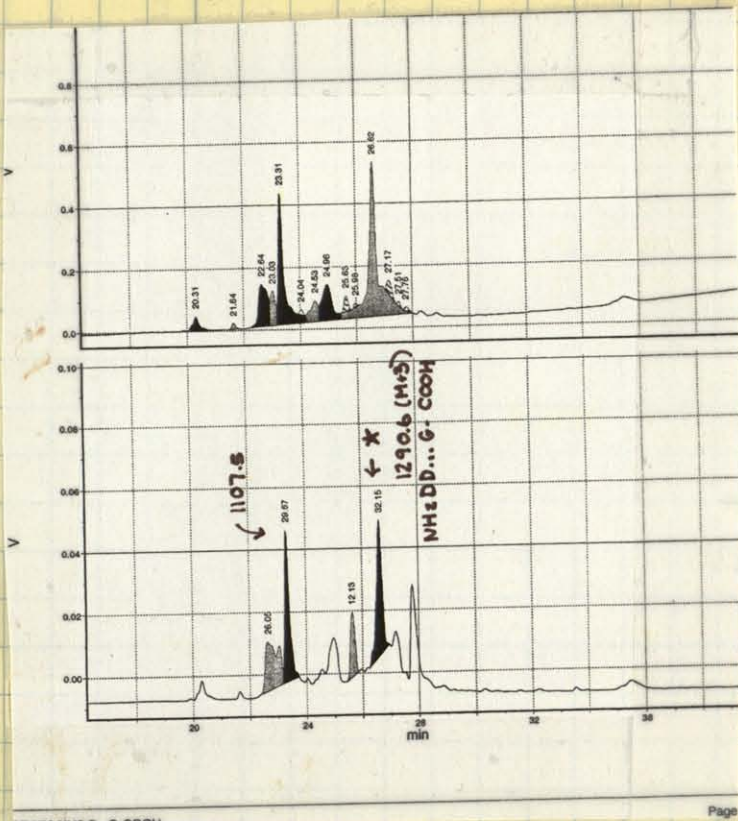
Mariner Mass Spectrum
D:_V020403_FmocSHI_G_25p7001.dat
Acquired: Feb 04 09:03:00 2003

His
Fmoc-Ser-Ile... Gly-COOH
mw by pepcalc: 2617.91
M+2 → 1309.96 M+3 → 873.64

- Perhaps should try HATU for tricky residues next time...
HATU/HOAT

9050 Completion of PAX (2-36)

Tuesday 2/04/03



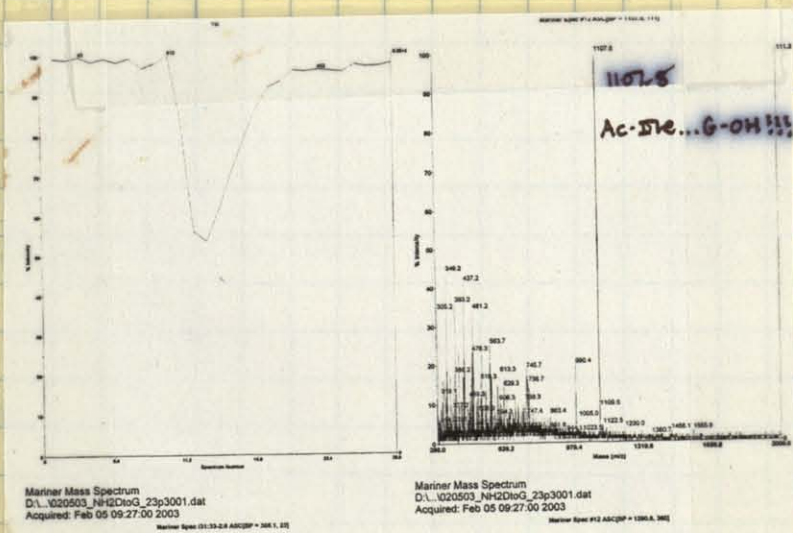
NH₂·DDL DALLADLESTTSHISK·
RPVFLSEETPYSYPTG·COOH

pepcalc. C₁₇H₂₆O₆N₄
mw 3869.22

M+3 → 1290.74 M+4 → 968.31

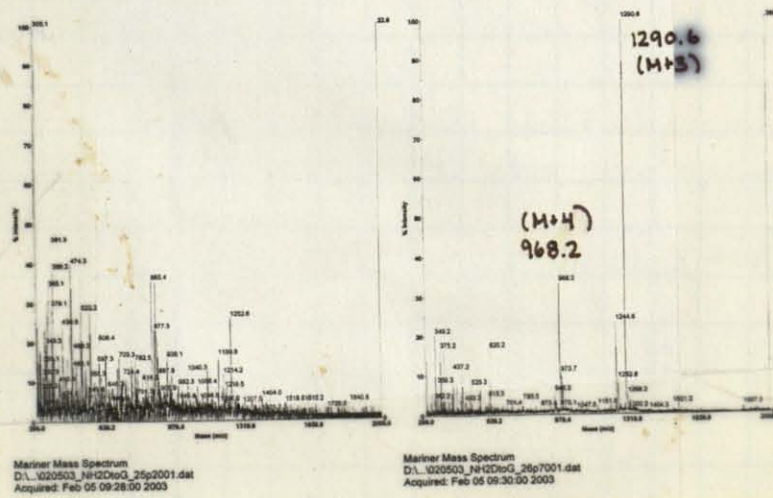
(so again seeing a significant peak w/ m/z 1107.5 (M+2) for Ac-ISK...G-COOH, so

even though coupling of His(Trt) and Ser were done by hand AND gave negative TNBS tests after coupling, a portion was still remaining uncoupled until capped after the T!!)



Mariner Mass Spectrum
D:_020503_NH2DioG_23p3001.dat
Acquired: Feb 05 09:27:00 2003

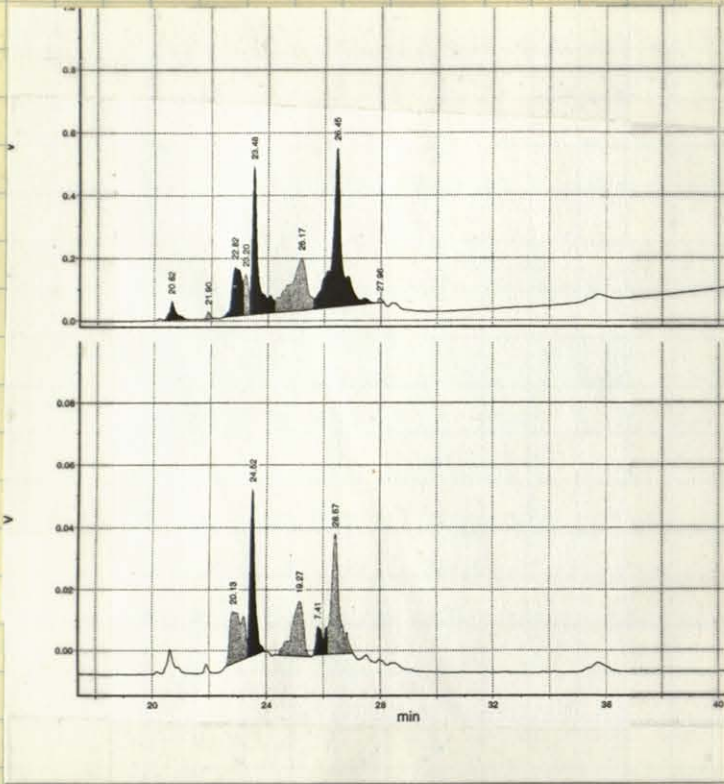
Mariner Mass Spectrum
D:_020503_NH2DioG_23p3001.dat
Acquired: Feb 05 09:27:00 2003



Mariner Mass Spectrum
D:_020503_NH2DioG_25p2001.dat
Acquired: Feb 05 09:28:00 2003

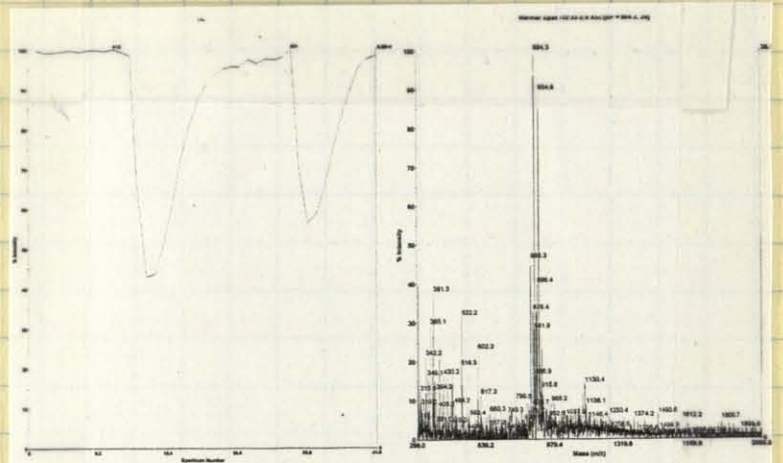
Mariner Mass Spectrum
D:_020503_NH2DioG_25p7001.dat
Acquired: Feb 05 09:30:00 2003

Met, His 4 Tag and Ac cap added by hand: Ac-His4-PAX(1-36)-OH 2/05/03



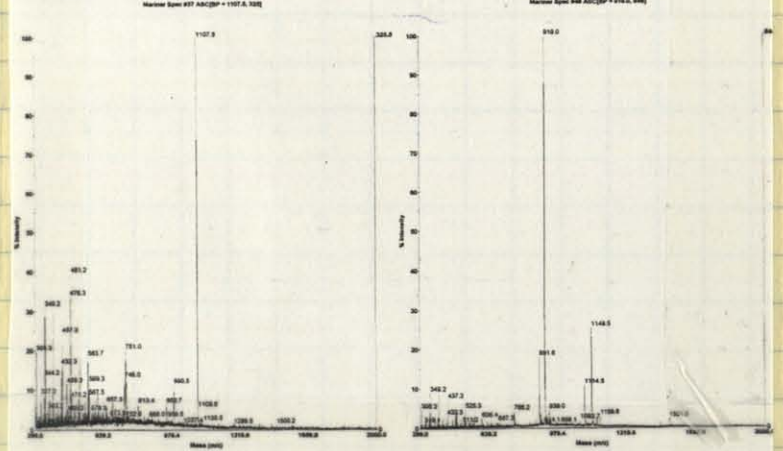
05/03 AcHHHH-Pax(1-36)-COOH

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Mariner Mass Spectrum
D:_AcHHHH-PaxOH_225p2001.dat
Acquired: Feb 06 09:27:00 2003

Mariner Mass Spectrum
D:_AcHHHH-PaxOH_225p2001.dat
Acquired: Feb 06 09:27:00 2003



Mariner Mass Spectrum
D:_AcHHHH-PaxOH_23p4001.dat
Acquired: Feb 06 09:25:00 2003

Mariner Mass Spectrum
D:_AcHHHH-PaxOH_23p5001.dat
Acquired: Feb 06 09:29:00 2003