

Table S1. Chemical shifts of 20 mM linear Aib-peptide, acetyl-GHIGPGR(Aib)FGGG-NH₂, at pH 5.0, 24 °C in 10% D₂O/90% H₂O.

Table S2. Chemical shifts of 20 mM Loop 5 at pH 5.0, 21 °C in 10% D₂O/90% H₂O.

Figure S1. HPLC chromatograms of (A) crude Loop 5 (analytical), (B) crude Loop 5 (prep), and (C) purified Loop 5 (analytical). Peak 1 is Arg-deleted Loop 5, [JHIGPG-(Aib)F(D-Ala)GZ]G-NH₂; Peak 2 is Loop 5. (A) Crude Loop 5 and (C) purified Loop 5 were analyzed on an analytical C18 column (Merck Lichrosorb, 4.6 x 25 mm, 10 micron) at 2 mL/min with a 0-60% AN gradient over 15 min. (B) Crude Loop 5 was purified at 8 mL/min on a preparative RP C-18 column (Cosmosil, 5C18-AR) with a 0-20% AN for 5 min, then 20-40% AN for 30 min, then 40-100% AN for 2 min followed by a return to 0.1% TFA/ H₂O. (D) MALDI mass spectrum of purified Loop 5.

Figure S2. HPLC chromatograms showing the progress of the chemical ligation of Loop 5/C (L) to MAPS core (MC) to form Loop 5-MAPS (LMC) from (a) 0 hr to (b) 2 hr. The reaction was monitored on an analytical RP C4 column (Vydac, 214TP54), eluting peptides at 2 mL/min with a gradient of 0-20% AN over 10 min followed by 20-32% AN over 30 min.

Figure S3. Mass spectra of (a) MAPS core (ES MS), (b) Linear 5/C-MAPS (ES MS) and (c) Loop 5/C-MAPS (MALDI MS).

Figure S4. Competition ELISAs of acetyl-RIHIGPGRAFY-NH₂ and acetyl-SIHIGPGRAFY-NH₂ for MAb 58.2 versus plate-bound rgp120. % antibody bound refers to the antibody bound to plate-bound rgp120.

Figure S5 Competition ELISAs of Loop 5 vs Loop 7 for MAb 58.2 versus plate-bound Linear 1. % antibody bound refers to the antibody bound to plate-bound Linear 1.

Figure S6. ELISA titration curves for Linear 5 (P2) and Loop 5 (P4-P6) antisera for rgp120. Background absorbance was subtracted.

Figure S7. Temperature coefficients for the amide protons of 20 mM linear Aib-peptide, acetyl-GHIGPGR(Aib)FG GG-NH₂, at pH 5.0 in 10% D₂O/90% H₂O. Temperature coefficients were determined from amide NH resonances obtained from 1D spectra at the indicated temperatures. Probe temperatures were determined from the chemical shift differences of a methanol standard according to the relationship reported by Van Geet, A. L. (1968) *Anal. Chem.* 40, 2227-2229.

Figure S8. Temperature coefficients for the amide protons of 20 mM Loop 7 at pH 5.0 in 10% D₂O/90% H₂O. Temperature coefficients were determined from amide NH resonances obtained from 1D spectra at the indicated temperatures determined by Van Geet's method (Legend, Fig. S7).

Figure S9. Portions of a 600 MHz ROESY spectrum of 20 mM (a) Loop 7 and (b) Loop 5 at pH 5.0, 10 °C. NOEs characteristic of β -turns at GPGR and RAibF(D-Ala/G) are indicated. The row projection is a shadow projection of the maximum data points along each column.

Table S1: ^1H Resonance Assignments for the V3 Linear Aib-peptide, Acetyl-GHIGPGR(Aib)FGGG-NH₂ at pH 5.0, 24 °C.

Residue	chemical shifts (ppm)					
	NH	αH	βH	γH	δH	Other
Acetyl						Ac ^{CH₃} 2.03
G1	8.25	3.86				
H2	8.44	4.71	3.20, 3.13		7.26	H ^{ϵ} 8.53
I3	8.22	4.21	1.84	1.44 1.14	0.89	δ^{CH_3} 0.83
G4	8.32	4.12, 4.03				
P5		4.41	2.22, 1.94	2.00	3.62	
G6	8.45	3.92				
R7	8.17	4.21	1.78, 1.70	1.59	3.18	NH ^{ϵ} 7.17
Aib8	8.34		1.29			
F9	7.83	4.57	3.21, 3.03		7.24	H ^{ϵ} 7.35, H ^{ζ} 7.30
G10	8.18	3.97, 3.91				
G11	8.07	3.97				
G12	8.34	3.92				
-NH ₂	7.56, 7.03					

Table S2: ¹H Resonance Assignments for Loop 5
[JHIGPGR(Aib)F(D-Ala)GZ]G-NH₂ at pH 5.0, 21 °C.

Residue	chemical shift (ppm)					
	NH	αH	βH	γH	δH	Others
J1		2.29	1.82	2.31	7.19	
H2	8.47	4.69	3.21, 3.11		7.24	H ^ε 8.54
I3	8.18	4.20	1.83	1.44, 0.88 1.14		δ ^{CH3} 0.83
G4	8.17	4.07, 4.00				
P5		4.45	2.25, 2.00	2.00	3.60	
G6	8.50	4.00, 3.87				
R7	8.17	4.19	1.83, 1.74	1.58	3.17	HN ^ε 7.16
Aib8	8.17		1.30, 1.18			
F9	7.70	4.60	3.22(β3), 2.95(β2)		7.21	H ^ε 7.34, H ^ζ 7.29
D-Ala10	7.96	4.38	1.36			
G11	8.18	4.47				
Z12		4.70				
G13	8.54	3.93				
-NH ₂	7.56, 7.03					

Figure S1

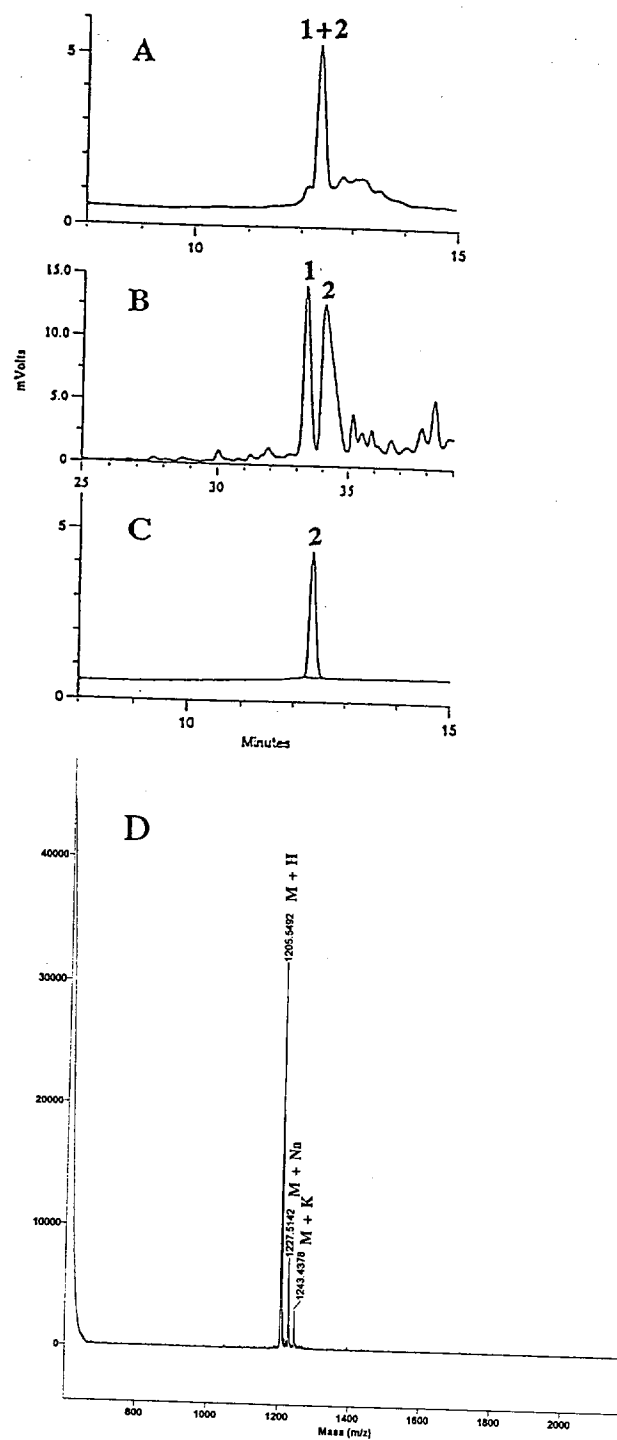


Figure S2

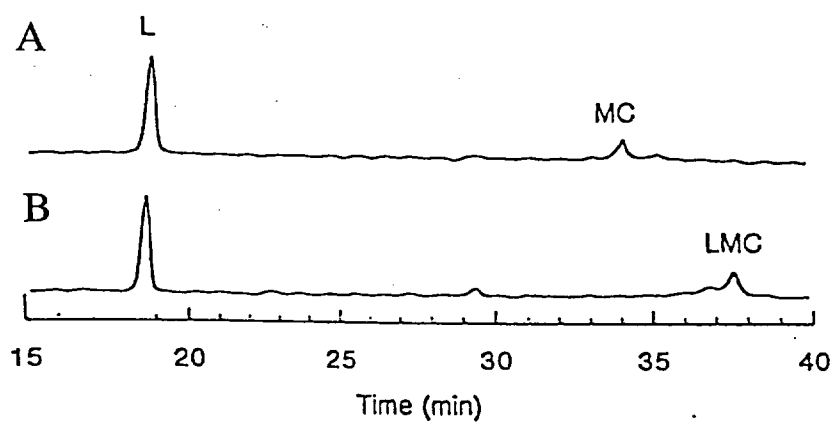


Figure S3

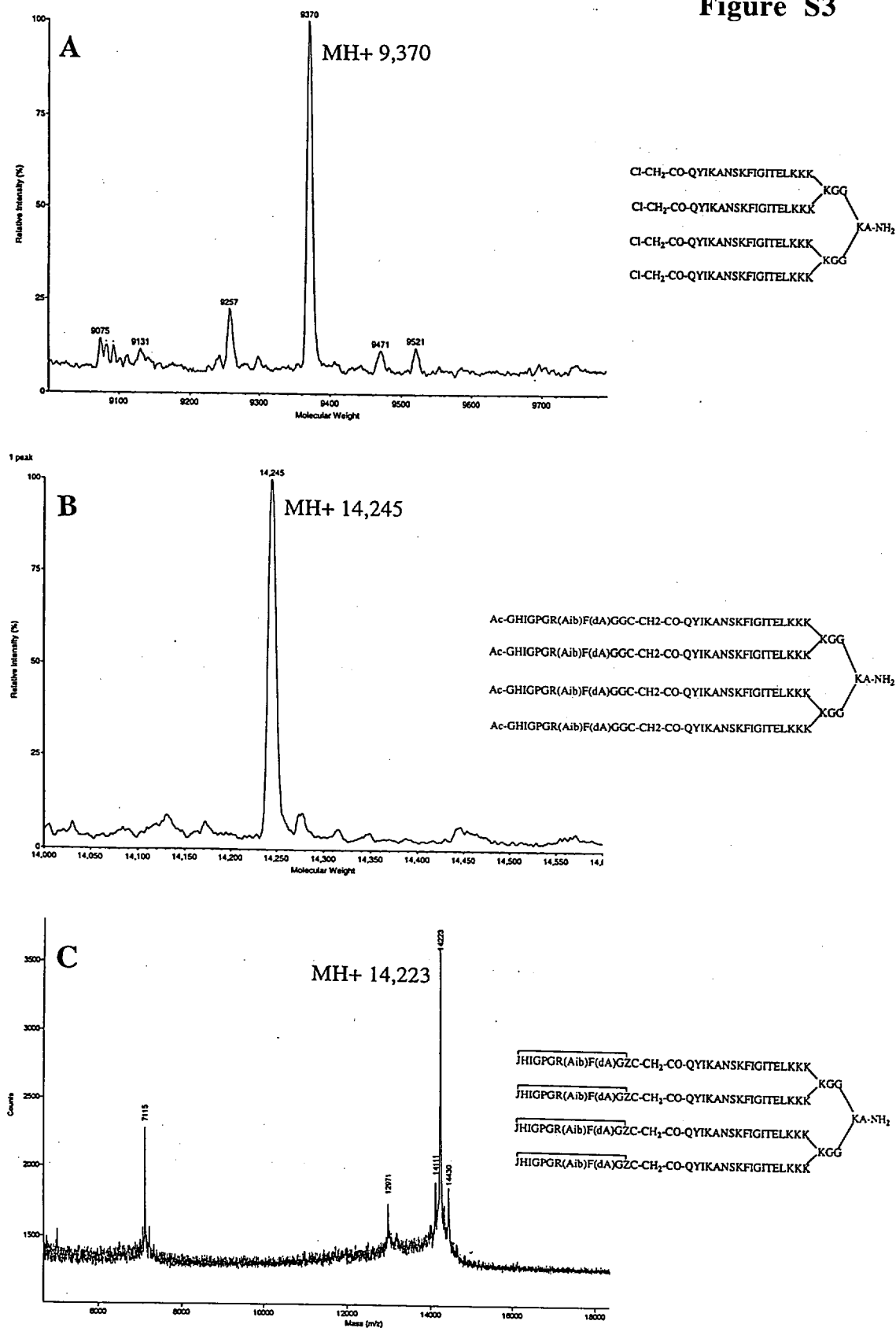


Figure S4

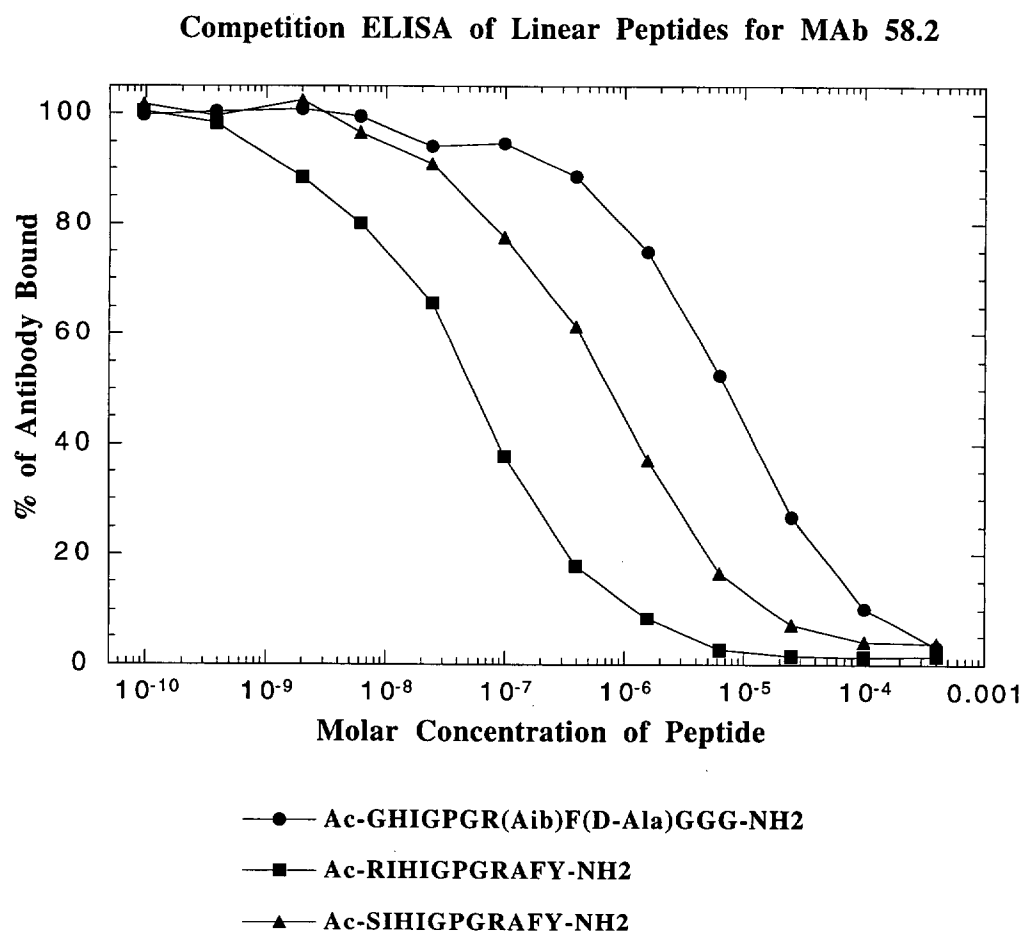


Figure S5

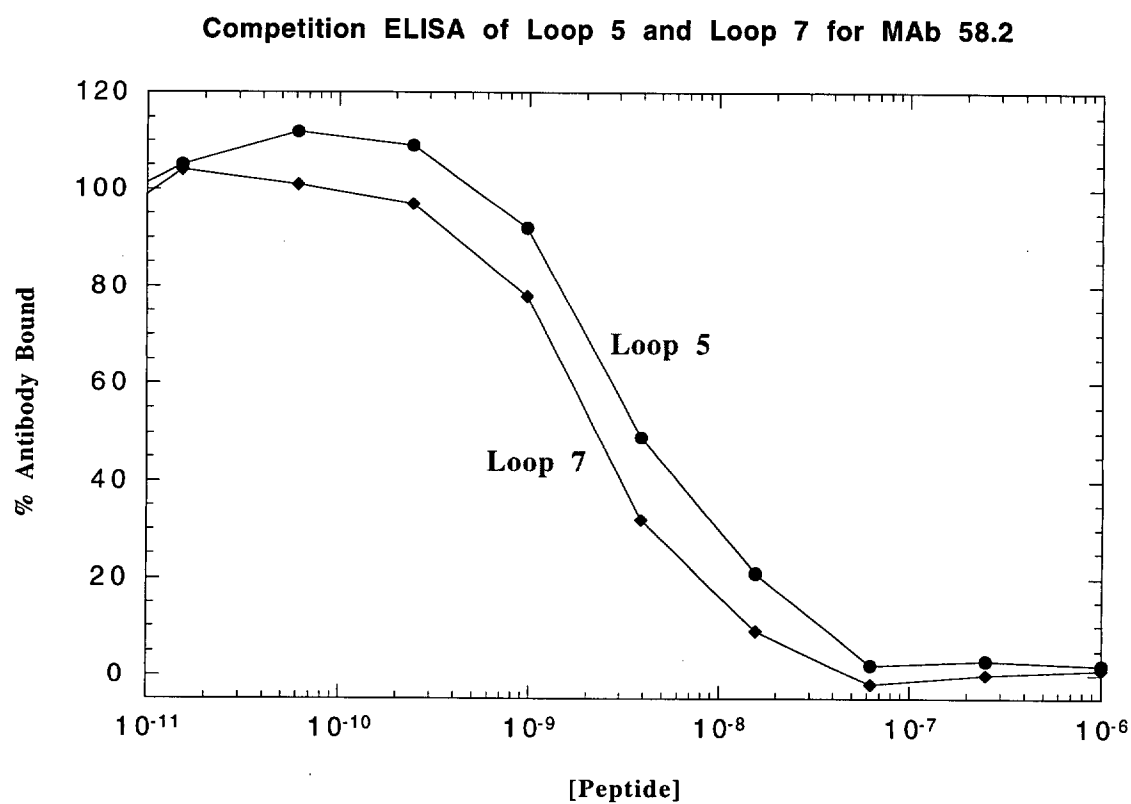


Figure S6

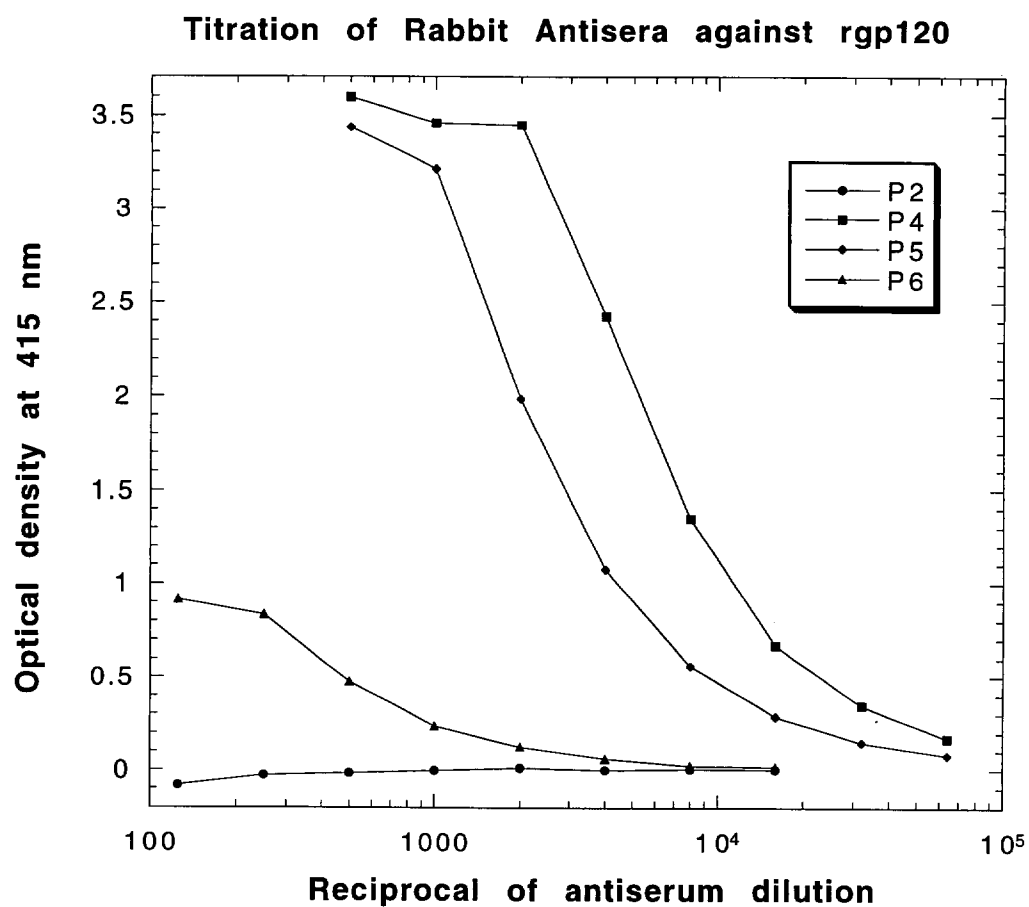


Figure S7

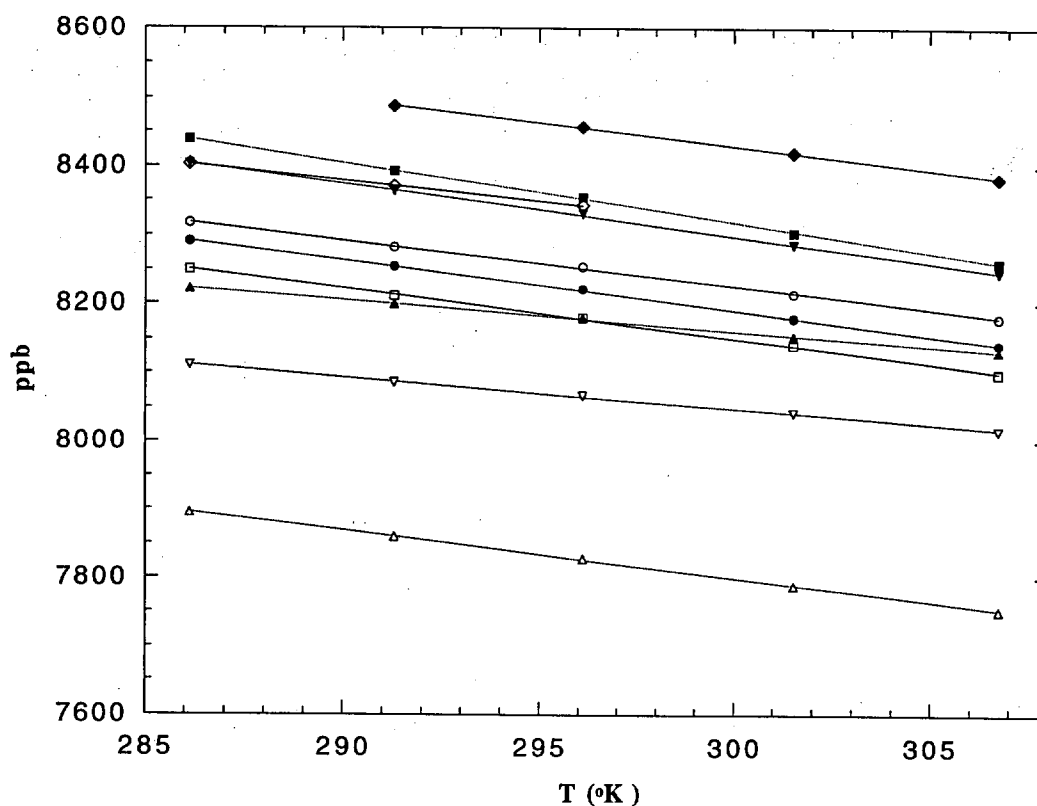
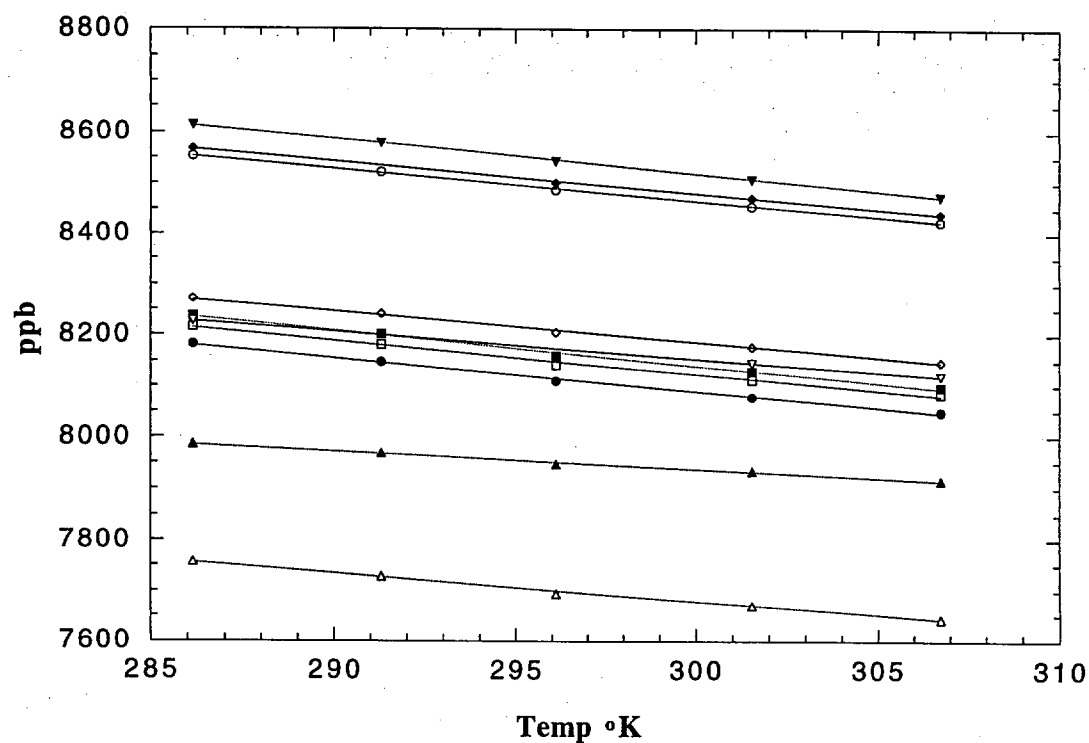


Figure S8



—○— H2	— $y = 10315 + -6.1625x$ R= 0.99934
—●— V3	— $y = 10043 + -6.5098x$ R= 0.99815
—◇— G4	— $y = 9976.2 + -5.9662x$ R= 0.99854
—◆— G6	— $y = 10313 + -6.1077x$ R= 0.9994
—□— R7	— $y = 10053 + -6.4314x$ R= 0.99751
—■— Aib8	— $y = 10175 + -6.7803x$ R= 0.99734
—△— F9	— $y = 9316.9 + -5.4598x$ R= 0.99785
—▲— dA10	— $y = 8969.2 + -3.4382x$ R= 0.99657
—▽— G11	— $y = 9729 + -5.2493x$ R= 1
—▼— G13	— $y = 10543 + -6.7468x$ R= 0.99964

Figure S9

