I. To calculate $T_2^*$, you measure the linewidth at half maximum intensity. $T_2^* = \frac{1}{\text{LWHalMax}^*}$

II. The $T_1$ is long for Peak A as it is not close to any other Hydrogens in the molecule while Peak B is close to the sugar like part of the molecule. The short $T_1/T_2$ would be some sort of CH resonance that is close to a lot of other Hydrogens, most likely in a sugar like molecule. A resonance with a moderate $T_1$ but short $T_2$ would be broad, presumably due to exchange, possibly an OH or NH or something coupled to N14.
The integrations do not match because a combination of longer T1s for the Carbons without attached Hydrogens, such as the carbonyl peak at 210 and presumably less H/C NOE. The quarternary Carbon that is not a carbonyl most likely integrates better as it will have more NOE from surrounding Hydrogens. The C-F Carbons will relax at a rate comparable to the CH Carbons as they can relax off of the Fluorine. However, by relaxing off of Fluorine, there is less NOE. The CHF Carbon is much less affected by this as it can relax off both the attached Hydrogen or Fluorine, whereas the other CF has no attached Hydrogen so integrates very low due to lack of CH NOE.

To increase the signal to noise for the CF Carbons, you would need to decouple them which would stimulate CF NOEs. This could potentially be done on the 500 instead of decoupling Hydrogen, but could be done on both nuclei on the VNMRS400. However, it is difficult to decouple both Fluorines as they are moderately far apart in chemical shift. Also, the 500 has about 2X overall better C13 signal to noise, so the experiment would be poorer for everything except the CF carbons.

You would need to increase d1 to probably 10-20 seconds and turn off decoupling during d1. This experiment would take a long time.
The peak at 47 is most likely broadened due to rotation of the aromatic relative to the sugar like part of riboflavin and coupling to quadrupolar N14. The peak at 86 is broadened somewhat due to coupling to Fluorine that through multiple bonds. The peak at 184 is presumably slightly broadened due to higher Chemical Shift Anisotropy and coupling to fluorine.

The instrumental setup reason is poor H1 decoupling, which leads to line broadening. The sample related reasons are paramagnetic species in the sample or molecule and coupling to quadrupolar nucleus.
4) The H1 spectrum shows nothing relevant to the F19 and P31 spectra and can be roughly ignored.

The F19 shows 3 peaks- 1 broad singlet, and 2 doublets. The downfield most peak is significantly truncated, indicating a long T2 relative to the acquisition time.

The P31 shows 2 peaks- the upfield peak is split into 7 lines, so that means 6 F19, so PF6. [there could be more lines but the signal to noise on the outer lines is already near baseline, but more Fluorines/Phosphorus does not make sense, so PF6 must be correct; also, integrating you get 1:6:15:20 which matches a septet]; the other peak is split into 3 lines, so PF2, the question would be whether the signal to noise is sufficient to be sure there are no more lines. It does not appear that there are more lines, and if you integrate, you can see that the middle line is twice as much as the outer lines, so 1:2:1 so a triplet (a pentet would be: 4:6:4 for the middle 3 lines; a septet would be 15:20:15)

The third F19 peak has no P31 coupling and no observed other coupling, probably just Fluoride ion (or a fluorine with no H1 or P31)

Integrating the PF6 in P31 spectrum:
1:56.28:127.5:165.2:133.2:55.6:9.7
Total = 548.5
PF2:
74.89:152.9:73.3
Total = 301.1
So Ratio PF6:PF2 in P31 = 64.5:35.5 = 1.82:1

Integrating the F19 spectrum:
One doublet is exactly 1.0:1.0, the other 6.73:6.82, so 6.78:1.

Which is PF2 vs. PF6? From the P31, the ratio is ~2:1 PF6:PF2, in the F19 it is 6.78:1, but the PF6 has 3X F19, so 2:1 * 3 ~ 6:1 so the peaks integrating to 6.73/6.82 are PF6, the other 2 PF2. Thus, the actual ratio of molecules in the F19 is 6.78/3:1 = 2.26:1

Alternatively, you could look up chemical shifts for each, or compare 1JFP, for the PF6 1JFP = 710 Hz, for PF2 1JFP = 934 Hz, so you could match the peaks that way also.

Thus, the approximate ratio is 2:1 but the problems with integrating such spectra the way they were acquired gives a skew of 1.8:1 for the P31 vs. 2.3:1 for the F19.

In the PF2 is 1 in the F19 spectrum, the F19 ion is 2.95, so including that in the ratio: 5.9:2.3:1 Fluoride:PF6:PF2

Problems with integration:
1) baseline flatness/background signal from TFA in F19 spectrum especially a problem for F19 ion; could fix with good baseline correction

2) Large sw/off-resonance effects especially for Fluorine; you can notice that the relative splitting integrations do not match Pascals triangle very well; off resonance effects would be one reason for that; again F19 ion would be affected; could fix by putting the center of the spectrum half-way between the peaks, so movetof; for the F19 ion you would probably have to collect to F19 data sets- 1 with the tof between the PF2 and PF6, the other between the PF6 or PF2 and the F19 ion

3) Truncation of the FID leads to poor resolution of peaks in F19, also implies at is too short; increase at or line broaden

4) T1s are probably at least moderately long, so d1 is too short in both spectra

5) signal to noise is a little low especially for the smallest lines; more scans

6) large splitting decreases signal to noise, could decouple F19 from P31 and vice versa; for P31{F19,H1} that could be done but only on new400
5)

The resonance is the N terminus of a peptide. (could be side chain arginine as well)

The N-terminus exchanges with solvent and rotates. The rotation must be fast on the NMR time scale to observe the resonance well, as it would be broadened due to rotational exchange. That is likely the case. The solvent exchange is affected by concentration, concentration of water, temperature, and pH. The sample was acidic, which helps in observing the exchangeable protons. With slower exchange and fast rotation, the NH2 would be observed, but would be observed as a singlet or a doublet with a small coupling to the Hα. The resonance is being observed as a triplet- a 1:1:1 triplet as if it had coupling to a quadrupolar nucleus, in particular a spin 1 nucleus, such as 14N. Normally, coupling to N14 is not observed due to the very fast relaxation of the N14 nucleus. However, the sample conditions were so acidic that the Nitrogen was protonated basically all of the time on the NMR time scale, so the Nitrogen is not trigonal. All quadrupolar nuclei are observed better in highly isotropic environment such as tetrahedral in contrast to the anisotropic of trigonal. Thus, the 14N relaxation is actually slow enough for the 1-bond 14N/1H coupling to be observed. The coupling constant is ~53 Hz, which is appropriate for a NH3 coupling, a 15NH is ~90Hz, a 15NH2 is ~80 Hz, a 15NH3 would be a little less, and 14N has a gyromagnetic ratio that is ~70% that of 15N.

Part B.
C13 would be split by N14 also, so a triplet, (plus a doublet to the Ha, and a quartet to the NH3+) if not H1 decoupled
N14 would be sharp and split by H3 resonance, so a quartet
N15 would be quartet, but with very poor sensitivity

Part C.

The peaks would be somewhat broader due to exchange, and might be suppressed by water suppression. Potentially, there would be slow enough exchange to see H-D coupling and isotope shift for NH2D, NHD2.
6) As temperature is increased the rotation around N-C bond on the NH2 increases, leading to a single NH2 resonance at higher temperature. Also, the H2O resonance shifts upfield as temperature is increased, and any resonance that exchanges with H2O also shifts, so the OH resonances and the NH2. DMSO briefly gets sharper as it tumbles faster at higher temperature; at the highest temperature it broadens somewhat, presumably due to either shimming or temperature instability. Some of the CH resonances shift very slightly presumably due to slight structural changes, and increased motion around the glycosidic bond and the conformation of the sugar ring. Slight changes are observed for the CH resonances, of ~0.01-0.03ppm, which could be related to structural changes caused by Hydrogen bonding, or rotation of the base or change in sugar conformation.

The 300 MHz and 600 MHz are different time scales, so the chemical shift between the resonances is the same in ppm, but half as much in Hz. The rotation is the same at the same temperature on both instruments, but how that relates to the frequency difference in Hz is different- what is fast at 300 is not fast at 600.

Also, note the chemical shift is the same at 45c on both instruments, but downfield at 38c (see Part A).

PART B.
The rotation of the NH2 is faster on adenosine than cytidine, presumably due to partial double bond in cytidine from resonance structures in contrast to adenosine, which does not have them, so it is a single bond.

PART C.
The rotation is slower in the RNA helix of the amino because it is Hydrogen bonded to the carbonyl of uridine, which slows the rotation. Thus, 2 separate shifts are observed for the amino. Also, the difference in chemical shift is much greater than in the free nucleoside because of the Hydrogen bonding and because of different ring current shifts from the base stacking above and below the adenosine in the helix.
7) Suppression of the water suppresses anything which exchanges with water on the NMR time scale. The OHs are suppressed as they exchange with water faster than the NH2 does at room temp. None of the peaks are completely suppressed because the T1 is short relative to the exchange rates so you can never completely suppress the OHs or NH2 by saturating water under these conditions. At higher temperature, you would see more suppression of the OHs and NH2 as the exchange rate increases without much change in T1. The gain value should be higher than in a normal 1D as the largest peak in the spectrum is no longer H2O, so the instrument can amplify the other signals more in the receiver/analog-to-digital converter without overflowing which leads to somewhat higher signal to noise.
8) The sample is 5-Fluorocytidine, and the nucleotide is β. To determine, you need to acquire 1D NOE. You should see NOEs from the H1’ to the H4’, H6 and H2’. The H2’ is on the opposite side but is not that far away in space as the angle between them is not perfectly trans. The H4’ NOE is what tells you that the H1’/H4’ are on the same side. At higher temp, you might get a little better data as the molecule tumbles faster in solution so you get further away from the NOE crossover point where NOE is 0. At 600 at 20C, it is very close to the crossover point, so there is little to no NOE or even somewhat negative NOE. Thus, at 600 the tumbling of the molecule in viscous solvent is slow relative to the magnetic field, while it is fast relative to the magnetic field on the 300 especially at 50C. In less viscous solvent, NOE would be positive at 600 for ~300MW molecule but not in DMSO/D2O; also positive at 50C on 600. Thus, for DMSO/D2O 1DNOE should be acquired at higher temperature, or should use ROE type experiment.
The D2O sample is TSP, so it has Na and Si 
Molecule 1 and has C13 spectrum 2 
Molecule 2 and C13 spectrum 1 

The correlation to C13 data: 
The sharp Boron peak means that its T2 is long, so that will cause the C13 to see Boron10 and Boron11 coupling; since B11 is spin 3/2, that leads to 2*I+1 lines = 2*3/2+1 = 4; since B10 is spin = 3, then 2*3+1 = 7. You could predict that would be the sharp Boron resonance because the Boron will be tetrahedral and has 4 equivalent groups attached to it, so highly symmetric which leads to sharp lines on quadrupolar nuclei.

The broader Boron peak means its T2 is shorter so the C13 sees an average coupling. However, it is not so fast that the C13 is sharp with no B-C coupling, it is intermediate so broad C13, not sharp and not split. You could predict that molecule 2 would fit that because it will be trigonal, so faster relaxation; overall a small molecule and somewhat symmetric on what the Boron is bound to, so not a super broad Boron peak, but moderately broad.

If you look closely at the C13 of the molecule 1, you will see that there is an isotope effect, so the B11-C13 chemical shift is not the same as the B10-C13 chemical shift. Also if you look real closely at the coupling constants, you will see that they are not the same but are actually the nearly exact ratio of the B11:B10 gyromagnetic ratio, ~50 Hz: 16.5 Hz in 1JCB and 3:1 in 
gyromagnetic ratio

For Silicon, you needed to have a long d1 and a number of scans to observe the peak, and dm = ‘nny’ as Silicon gyromagnetic ratio is negative. Silicon could also be done as a DEPT, that would help a lot. You could have also done HMBC for this. While the sample was moderately concentrated, the signal to noise for silicon is not that good- moderately low abundance and low gyromagnetic ratio, also no NOE, long T1... If the parameters were suboptimal, then you probably observed nothing.

Boron and Sodium are both quadrupolar, so shorter acquisition time, and short d1 could help you see the resonances faster, although both are sensitive enough that it does not matter much. For Boron, the large peak from the glass gets in the way of observing real peaks, but the real peaks are either very sharp relative to the glass or moderately sharp relative to the glass. Since the glass a longer T1 than the real peak, you could improve the spectra by using a large number of ss to saturate it, before acquiring real data. Also, acquiring data with an empty NMR tube or a tube with a sample an no Boron (so the CDCl3 standard would work), you can see the glass peaks and compare to the spectra that you get.

Chloroform:
The solvent is mostly CDCl3, but there is some residual CHCl3. The small peak is the CHCl3 peak, and there is an isotope shift of about 0.2 ppm between H1 and H2. The CHCl3 peak is a singlet as the H1 is decoupled. The CDCI3 peak is a triplet as deuterium is spin = 1, so 2NI + 1 = 2*1*1+1 = 3. The ratio is 1:1:1 as a spin = 1 triplet is 1:1:1.

There are a large number of lines that could be observed but are not because of T2 relaxation issues. The 2 separate reasons are isotope shift from the chlorine as in Part A, and quadrupole splitting from chlorine as in this part to Deuterium.

Both Cl35 and Cl37 are spin = 3/2, so 2NI + 1 = 2*3*3/2 + 1 = 10. The ratio would be: 1:3:6:10:12:12:10:6:3:1 for both Cl35 and Cl37
You do not see these lines as the chlorine relaxation is fast on the NMR time scale so the carbon sees an average, so just one line to chlorine. You do observe the splitting to the quadrupolar Deuterium, but Deuterium relaxation is much slower than Chlorine. The difference relative to the Boron is that Cl35 or 37 are both much shorter T2 than either Boron, so there is not much broadening observed for CDCl3 or CHCl3. The Deuterium is comparable to the tetrahedral Boron, so you do see splitting. The isotope shift of CHCl3 relative to CDCl3 is bigger than the Boron shift, as H is smaller than B.

The other possibility is to observe isotope shift from the chlorine. This isotope shift would be more complicated than in Part A as there are 3 Chlorines, so you could have 3 Cl35s, 3 Cl37s, 2 Cl35s and 1 Cl37, or 1Cl35 and 2 Cl37s, so instead of 2 lines as in Part A, there would be 4. Instead of a ratio of 3:1, the ratio would be: 27:27:9:1 [Cl35:2Cl35/1Cl37:1Cl35/2Cl37:Cl37]. The isotope shift is not observed because the Carbon line is too broad to observe them. The shift is only ~0.01 ppm, but the linewidth at half height is ~0.04 ppm. While acquisition/processing/shimming parameters might not have been optimal to observe the isotope effect, the primary reason for not observing it is the line width/T2 of CDCl3. The Carbon resonance is broad relative to Part A because the Carbon on CDCl3 is coupled to 4 quadrupolar nuclei. While the coupling to the 3 Chlorines is not observed, the coupling is still there and induces more rapid relaxation of the Carbon. In addition, the coupling to Deuterium also increase the relaxation of the Carbon due to the quadrupole effect. The difference in Part A was that carbon was bonded to 2 spin = 0 nuclei (Carbon), 1 spin = ½ (Hydrogen) and the 1 Chlorine, so T2 relaxation of it was much slower than CDCl3, so sharper line, more observable. In addition the large number of potential lines also broaden the observed resonance for CDCl3.

The total number of possible lines would be:
4 (deuterium splitting + CHCl3(if H1 decoupling on)) * 10 (chlorine splitting) * 4 (isotope shift chlorine)  = 160.

If the Cl35/C13 coupling were different than Cl37/C13, I think it would be 240 (the CD/HCl35(3) or CD/HCl37(3) would have 40 lines, the CD/HCl35(2)Cl37(1) and CD/HCl35(1)Cl37(1) would have 80 each.
If H1 decoupling were turned off, then $5 \times 10^4 = 200$ or $280$ with different Cl/C coupling constants.