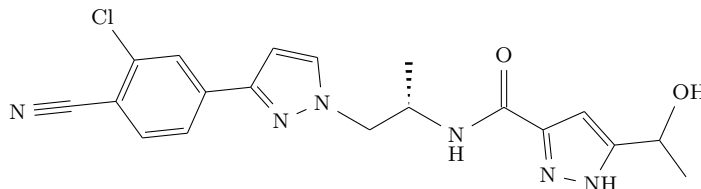


3 Peak Assignments

3/6: Darolutamide is an anti-androgen compound used in the treatment of prostate cancer in older men.

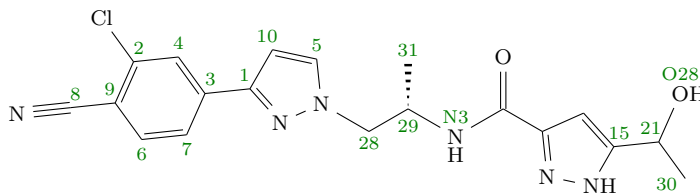


A single 300 mg dose tablet weighs approximately 610 mg. A sample of darolutamide was produced by crushing a single tablet, adding approximately 1.5 mL of d₆-DMSO, mixing thoroughly, and then spinning the mixture in a microfuge at $10\,000 \times g$ for 10 min to separate the liquid phase. This solution was used to generate the data in the **unknown-D_5.46_2025** dataset. The experiments in the dataset are as follows.

1. ¹H 1D.
2. ¹³C 1D.
4. DEPT 135 (¹³C).
5. ¹H COSY.
6. ¹H-¹³C HSQC.
7. ¹H-¹³C HMBC.
8. ¹H-¹⁵N HSQC.

The sample is clearly a mixture of the known compound (darolutamide) and excipients/fillers from the tablet. Use everything you know about NMR so far to identify the proton and carbon signals that correspond to the drug to the best of your ability. Your final result should be a structure with the carbons labeled according to the ¹³C signals in the spectrum, where possible from the data, starting with the carbon at 147.8 ppm labeled as carbon #1, and a table of proton/carbon correlations with proton chemical shifts and multiplicities, similar to what we did in class with adenosine. Indicate where you used the COSY and HMBC data to support your conclusions.

Answer. To the best of my ability, here are the assignments for the darolutamide portion of the provided data.

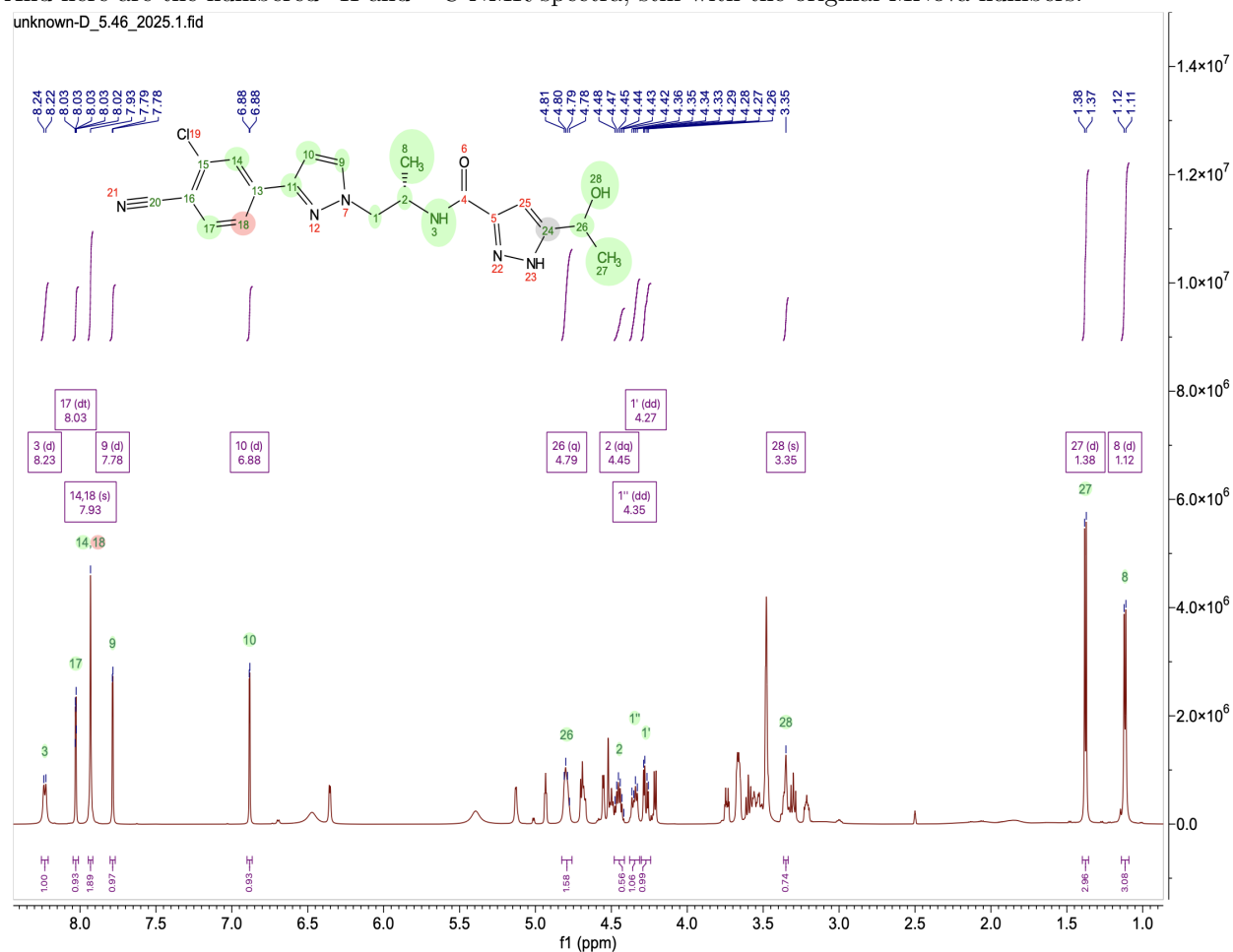


Here is the desired table.

MNova #	Carbon #	¹³ C (ppm)	DEPT	¹ H (ppm)	¹ H Multiplicity	COSY	HMBC
11	1	147.46	Quat				4,5,6(w),7,10
15	2	139.63	Quat				4,6,7
13	3	135.98	Quat				4,6,7
14	4	135.05		7.93	s		
9	5	133.24		7.78	d	10	10,28
17	6	125.77		8.03	dt	7	
18	7	124.20		7.93	s	6	6
20	8	116.36	Quat				4,6(w),7(w)
16	9	110.00	Quat				4,6,7
10	10	104.25		6.88	d	5	5
24	15	81.44					O28
26	21	73.36		4.79	q	30,O28	
1	28	55.81	CH ₂	4.27,4.35	dd,dd	29	5,29,31,N3
2	29	44.92		4.45	dq	N3,28,31	28,31,N3
27	30	23.87		1.38	d	21	
8	31	18.07		1.12	d	29	28,29,N3
3	N3			8.23	d	29	
28	O28			3.35	s	21	

And here are the numbered ¹H and ¹³C NMR spectra, still with the original MNova numbers.

unknown-D_5.46_2025.1.fid



unknown-D_5.46_2025.2.fid

