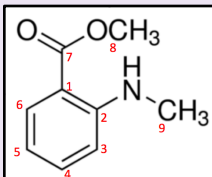


NMR Assignments for Dimethyl Anthranilate

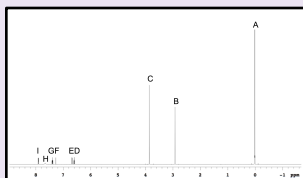
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Dimethyl anthranilate (DMA) is used to give grape flavoring to Kool-Aid, sodas, gum, and other food products. DMA is a di-substituted benzene ring with an ester and a secondary amine. Its chemical formula is $C_9H_{11}NO_2$, and its molecular mass is 165.21 g/mol. The sample was dissolved in $CDCl_3$ with the reference molecule tetramethylsilane (TMS). The DMA concentration in the sample was 100mM. All data were recorded at 500 MHz (1H).

1H 1D NMR Spectrum

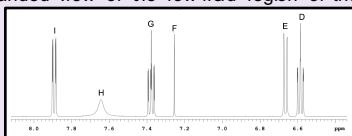
The proton spectrum is shown below with a table that associates each resonance with its chemical shift. The spectral width was 5319.1 Hz



Peak	Chemical Shift (ppm)
A	0
B	2.91
C	3.85
D	6.59
E	6.67
F	7.26
G	7.37
H	7.64
I	7.89

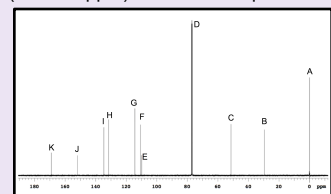
(10.636 ppm) and the acquisition time was 3.080 sec. Resonance A at 0 ppm is from the reference compound TMS. The H8 and H9 protons have a greater electron density than the other protons, so they are resonances B and C. Since nitrogen has a lower electronegativity than oxygen, H9 would be anticipated to be more upfield. Resonance B is assigned to H9 and resonance C is assigned to H8.

The figure below is an expanded view of the low-field region of the proton spectrum at the The solvent $CDCl_3$ has a small amount of $CHCl_3$, which is typically has a chemical shift near 7.26 ppm. Thus, resonance F is from the solvent. Uniquely broad resonances are characteristic of protons bound to ^{14}N . Resonance H is assigned to the proton bonded to the nitrogen.



^{13}C 1D NMR Spectrum

The spectral width used to record the ^{13}C spectrum was 25510.2 Hz (202.85 ppm) with the acquisition time of 1.28451 sec. The TMS

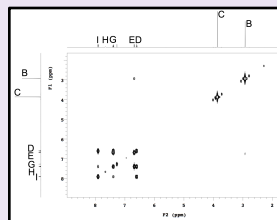


Peak	Chemical Shift (ppm)
A	0
B	29.6
C	51.4
D	77.0
E	110
F	111
G	114
H	132
I	135
J	152
K	169

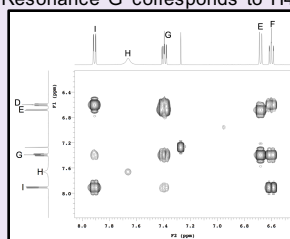
resonance at 0 ppm is labeled A. The chloroform resonance at 77 ppm is labeled D. As we predicted for the 1H spectrum, C8 and C9 are the two carbons with the highest electron density and because nitrogen is less electronegative than oxygen, C8 is more deshielded. Therefore, resonance C is C8 and resonance B is C9. C7 is bonded to two oxygens. It is the most deshielded carbon, so resonance K is assigned to C7.

COSY NMR Spectrum

The COSY spectrum is a map of J-coupling interactions between protons. The data shows H9 has a correlation with resonance E. The closest proton, which is five bonds away from H9, is H3. This is the only possible proton for this correlation, so resonance E corresponds with H3. Resonance E, H3, also has coupling with resonance G.

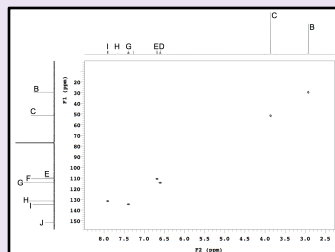


Resonance G corresponds to H4. Resonance G, H4 has a strong coupling interaction with resonance D and a weaker coupling interaction with resonance I. Since we anticipate the three bond coupling to be stronger than the four bond coupling, we assign resonance D to H5 and resonance I to H6.



HMQC NMR Spectrum

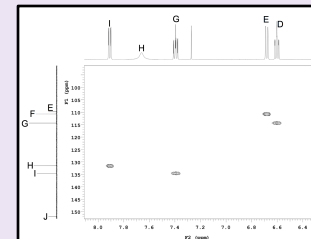
The HMQC spectrum maps scalar coupling correlations between directly bound 1H and ^{13}C nuclei. The correlation with the ^{13}C



resonances B and C and the 1H resonances B and C respectively confirms the proposed assignments. Resonance B is associated with C8 and H8, and resonance C is associated with C9 and H9. The ^{13}C resonances E and J do not correspond with any protons, so they must be the tertiary carbons, C1 and C2. As C7 is the most downfield resonance K, C1 would have more electron density and would be upfield from C2. The downfield resonance J corresponds with C2 and upfield resonance E corresponds with C1.

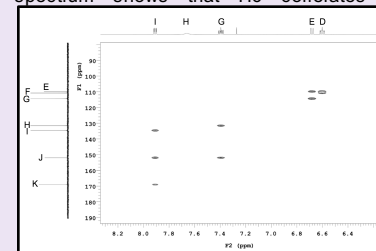
The figure to the right is an expanded view of the HMQC spectrum showing the aromatic region. The 1H resonance E corresponds with H3 and correlates with the ^{13}C resonance F, so resonance F corresponds with C3. The 1H resonance I, H5, correlates with

the ^{13}C resonance H. Resonance H is assigned to C5. The 1H resonance G, H4, correlates with the ^{13}C resonance I. The 1H resonance D, H6, correlates with the ^{13}C resonance G, so resonance G corresponds with C6.



HMBC NMR Spectrum

The HMBC data maps the hydrogen and carbon interactions across two and three bonds. The spectrum confirms the proposed assignments. As H6 is expected to interact with C1, 5, 7, 4, and 2, the spectrum shows that H6 correlates with C7, C2 and C4.



Likewise, H4 should correlate with C3, 5, 2, and 6 and does correspond with C2 and C6. H3 should interact with C2, 4, 5, and 1, and it does correspond with C1 and C5. Lastly, H5 should associate with C4, 6, 1, and 3, and H5 does correspond with C1 and C3. None of the second carbon associations were observed at this contour level, as they are weaker than other associations.

Concluding Table

The table below summarizes the data of each chemical shift to the assignments of carbons and hydrogens.

	1	2	3	4	5	6	7	8	9	N	$CDCl_3$	TMS
^{13}C Peak (ppm)	E	J	F	I	G	H	K	B	C	-	D	A
1H Peak (ppm)	110	152	111	135	114	132	169	29.6	51.4	77.0	0	0
	-	-	E	G	D	I	-	B	C	H	F	A
			6.67	7.37	6.59	7.89		2.91	3.85	7.64	7.26	0

References

Claridge, Timothy D. W. *High-Resolution NMR Techniques in Organic Chemistry*. Amsterdam: Elsevier, 2009.

Spectral Database for Organic Compounds SDBS. National Institute of Advanced Industrial Science and Technology (AIST). Japan. Web. 3 October 2017. <http://sdb.sdb.aist.go.jp/sdb/cgibin/dir/ect_frm_me_t_op.cgi>