



Chem115 Fall 2023 - Ilana Berlin/Lab7: Nuclear Magnetic Resonance (NMR) Spectroscopy I: Simple Structure Determination/LabReport7

Jason Cody - Oct 29, 2023, 6:48 PM CDT

Assignment #14 - LabReport7

 You cannot edit this entry after it is graded.

Description Proton NMR Spectra

I worked in a group with

The work for this assignment My notebook
is in

Grade **9 / 10**

Graded on Oct 29, 2023, 6:48 PM CDT

Jason Cody - Oct 01, 2021, 10:36 AM CDT

TITLE: (Insert experimental title here. All italicized text in parentheses should be followed and then deleted throughout this template).

Purpose: (Insert experimental purpose here).

Reference: Kanley, L. J., *Introduction to Chemistry in the Laboratory*, 20th Ed., Lake Forest College, 2021. Experiment 25, Appendix 2a. (Edit the experiment title and/or appendix letter; add other references, if used, following the same format.)

Observation and Data: (Write your observations, conclusions, and/or tables and figures here. If needed, insert tables and edit the header: Table 1, Preparation of Standard Solutions. If needed, insert figures and edit this caption below the figure: Figure 1, Beer's Law Plot of B12 Standard Solutions at $\lambda = 530$ nm. Number tables and figures in order of appearance in the report.)

Calculation: (Insert sample calculation here, if relevant. Otherwise, delete this section entirely).

Conclusion: (State the quantitative values (percent error and/or CV) to indicate how well the goals of the experiment have been met; answer any questions in the experiments' instructions, too).

ReportTemplate.docx (15.5 kB)

Jason Cody - Sep 30, 2020, 2:14 PM CDT

Date and Title

Ilana Berlin - Oct 23, 2023, 11:23 AM CDT

10/23/2023

Jason Cody - Sep 30, 2020, 2:14 PM CDT

Purpose

Jason Cody - Oct 29, 2023, 6:43 PM CDT

Unknown organic compound samples the more general "two samples" is sufficient here (if you give the numbers, that indicates that you wrote the purpose after you started--not the goal here! 30 and 33 were scanned by a 60 MHz proton NMR. By comparing estimates What is estimated/predicted? and values from the NMR, we can make an educated guess as to which organic compounds the unknown is. How many samples are predicted?

Jason Cody - Sep 30, 2020, 2:14 PM CDT

Reference

Jason Cody - Oct 29, 2023, 6:44 PM CDT

"MilliporeSigma | United States." www.sigmaaldrich.com, 2022, www.sigmaaldrich.com/US/en. Accessed 22 Oct. 2023. Good.

Kateley, L. J., *Introduction to Chemistry in the Laboratory*, 20th Ed., Lake Forest College, 2021, Experiment 7 Appendix C,G. Good.

Ilana Berlin - Oct 23, 2023, 3:08 PM CDT

Data and Observations

Jason Cody - Oct 29, 2023, 6:47 PM CDT

Predictions were made for 10 organic compounds. (Figures 1 and 2) Unknown compounds 30 and 33 were run through Anasazi 60 MHz FT-NMR and the resulting graphs were printed. Data from NMR was compared to predicated data and samples were identified. (Figure 3 and 4). What happened to the double bonds in your C6 rings? Next time, please put your shifts in order (a, b, c, etc.) of increasing chemical shifts. This way, when they are read off of the spectrum, they'll match more easily (your table is jumbled).

TABLE 1: STRUCTURES AND NMR PREDICTIONS, compounds 1-5 (no spin-spin splitting)

| Name | Condensed Line Formula | Complete Lewis Structure (All atoms must be shown) | # of signals | Estimated chemical shift | Proton ratio |
|-------------------------------|------------------------|---|--------------|--------------------------|--------------|
| mesitylene acid | $(C_6H_5)(CH_3)_3$ | | 2 | 2.3 6.5-8 | 9 3 |
| methyl acetate acetate | CH_3COOCH_3 | | 2 | 2.0 3.7 | 3 3 |
| 1-bromopropion- acolone | $(CH_3)_3CCOCH_2Br$ | | 2 | ~0.9 4.6 | 9 2 |
| tert-butyl methyl ether | $(CH_3)_3COCH$ | | 2 | ~0.9 3.3 | 9 3 |
| dimethoxy- benzene | $C_6H_4(OCH_3)_2$ | | 2 | 4.5-8 3.8 | 4 6 |

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Figure 1. Compounds 7,9,1,2 and 3

TABLE 2: STRUCTURES AND NMR PREDICTIONS, compounds 6-10 (no spin-spin splitting)

| Name | Condensed Line Formula | Complete Lewis Structure (All atoms must be shown) | # of signals | Estimated chemical shift | Proton ratio |
|-------------------------------|-------------------------|---|--------------|--------------------------|--------------|
| pinacolone | $(CH_3COC(CH_3)_3$ | | 2 | 2.2 ~0.9 | 3 9 |
| di-tert- butylben- zene | $(C_6H_5)[C(CH_3)_3]_2$ | | 2 | ~1.29 6.5-8 | 18 4 |
| malonic acid | $CH_2(COOH)_2$ | | 2 | 9.12 2.2 | 2 2 |
| p-xylene | $(C_6H_5)(CH_3)_2$ | | 2 | 2.3 6.5-8 | 6 4 |
| methoxy- aceton- itrile | CH_3OCH_2CN | | 2 | 4.8 3.3 | 2 3 |

2

Figure 2. Organic compounds 4,5,6,10 and 8

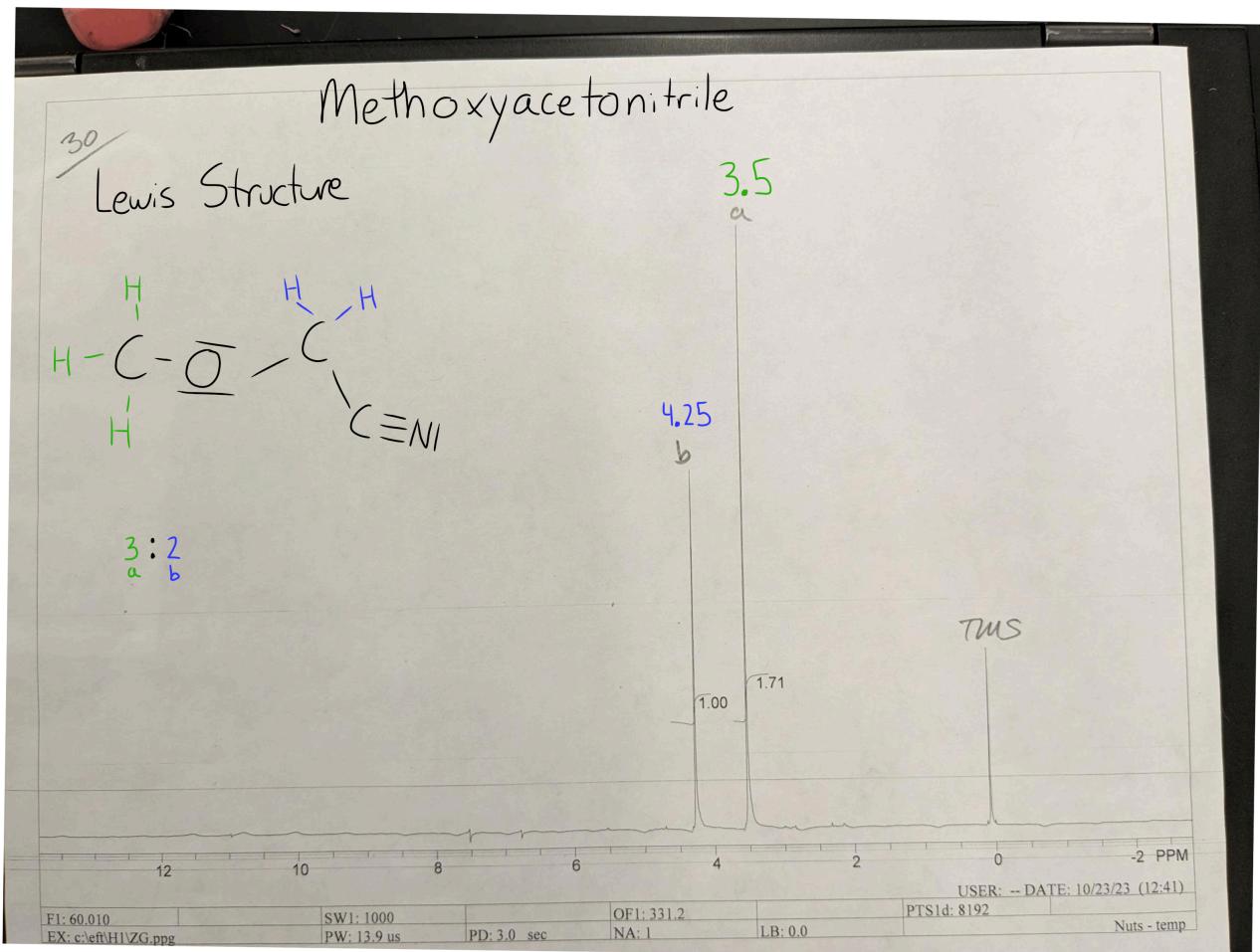


Figure 3. Sample 30 - Methoxyacetonitrile Good.

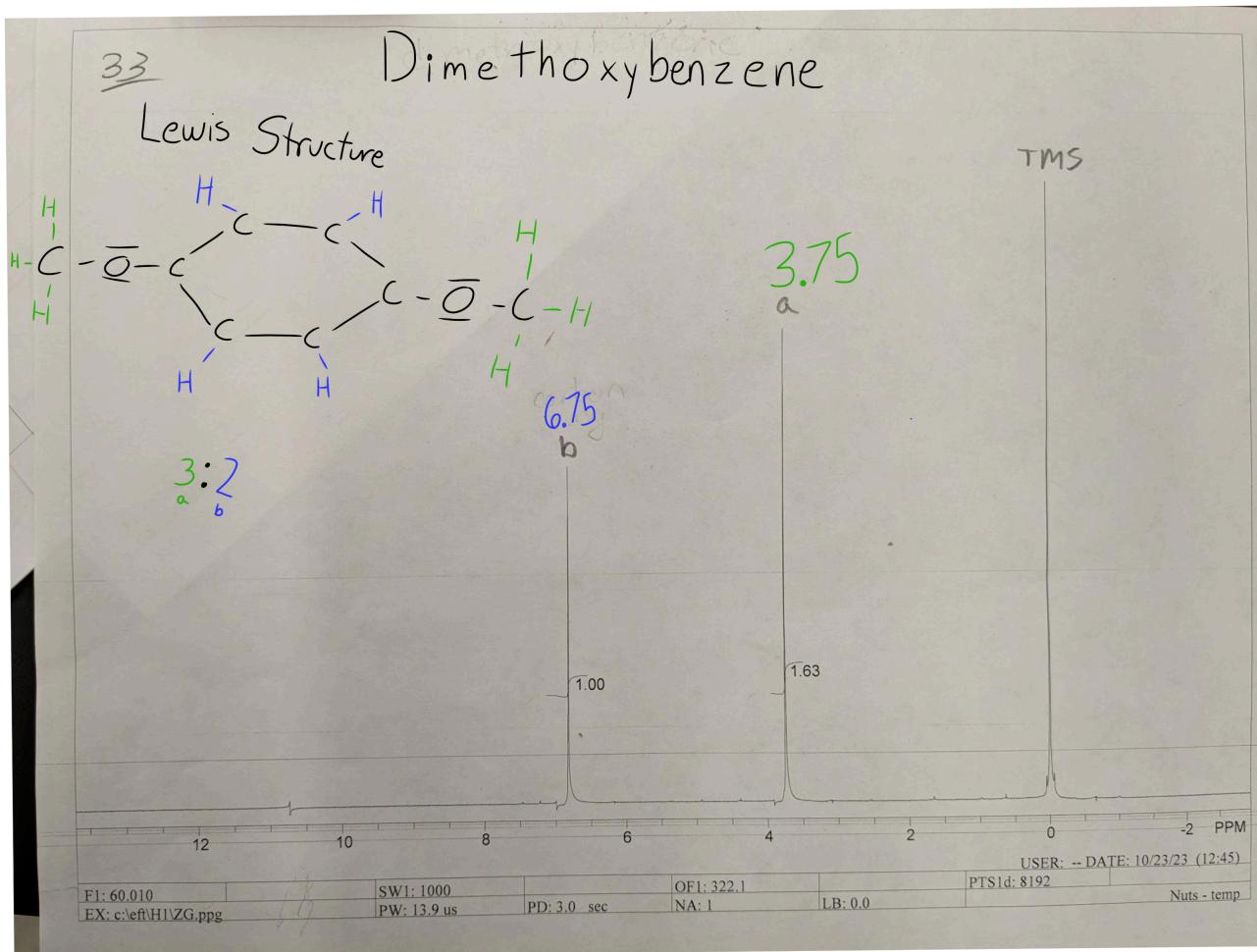


Figure 4. Sample 33- Dimethoxybenzene Good, except for the C atoms with only 3 bonds!

Jason Cody - Sep 30, 2020, 2:14 PM CDT

Calculations

Jason Cody - Sep 30, 2020, 2:14 PM CDT

Conclusions

Jason Cody - Oct 29, 2023, 6:48 PM CDT

Sample 30 is likely Methoxyacetonitrile and sample 33 is likely Dimethoxybenzene. All organic samples had 2 electron proton NMR signals. Estimated chemical shift changes based on shielding from other elements near the proton

(hydrogen). The size of the peaks on the NMR graph shows the relative proton ratio. What about the relative shifts of a nearby O or Br atom? a ring?