

Identification of an Aldehyde or Ketone by Qualitative Analysis.

Reading Pavia (5th edition) Experiment 52D, Appendix 1 (Tables of Unknowns and Derivatives), Appendix 2 (Procedures for Preparing Derivatives)

General

Using modern spectroscopic techniques (NMR, IR, MS), the structure of nearly any organic molecule can be determined. How was this done before these techniques were available? One type of analysis (that even predates IR spectroscopy) is qualitative analysis. Functional groups in a molecule can be “detected” by noting the behavior of the molecule under certain test reaction conditions. A key part of this type of analysis involves the formation of a solid derivative of the functional group and the determination of its melting point. Comparison to literature data for known compounds can yield the identity of the original compound. This is the basis for the current experiment.

Procedure

You will start by analyzing known aldehydes and ketones using the following standard test conditions listed in Experiment 52D. Follow the procedure in the text for each of these using the test compounds listed in the table below (next page):

2,4-dinitrophenyl hydrazine (2,4-DNP) test
Tollen’s test
iodoform test
ferric chloride test
(we are omitting the chromic acid test!)

You will then be given an unknown aldehyde or ketone. Run only the necessary tests to determine whether it is an aldehyde or ketone (and if it is a ketone, whether it is a methyl ketone), then you will make two solid derivatives of the compound, the 2,4-dinitrophenyl hydrazone and the semicarbazone. The procedures for making these are in Appendix 2 of Pavia.

Finally, through measurement of the melting point (or boiling point) of the original compound and the melting point of the formed derivatives, you will be able to find the identity of the compound. Once you have conclusively determined of the aldehyde or ketone you will perform spectroscopic analysis to verify it.

Prelab

In your notebook have the usual **Name**, **Date**, **Title**, **Purpose**, and show an **Outline** of the procedures for each test. Show a table in your book that lists the name and structure of each of the known aldehydes and ketones, along with the mp (for solids) or bp (for liquids). You can find this information in Appendix 1 in Pavia.

Also prepare a table like the one shown on the next page.

Test	Test Compounds	Results
2,4-DNP	cyclohexanone p-anisaldehyde benzophenone	
Tollens test	acetone benzaldehyde cyclohexanone butanal	
iodoform	2-heptanone 3-heptanone 2-pentanol	
ferric chloride	2,4-pentanedione	

To Complete the Experiment – Partial Report

Summarize the results from your analysis of the unknown compound and clearly indicate how you determined its identity. Be sure to note any ambiguous results or observations from the known or unknown compounds. Include the analysis of your NMR spectra in your notebook and turn in a copy of your original spectra.