

Identifying an Unknown Aldehyde or Ketone

Please see Experiment 10: Identifying an Unknown Aldehyde or Ketone in the Lab Manual for background and reference.

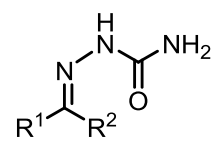
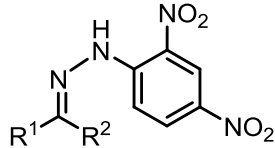
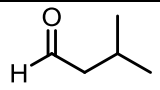
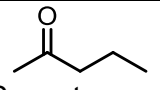
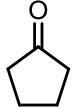
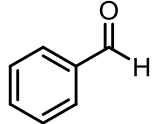
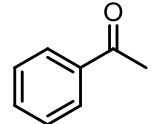

In this lab, you will use a combination of modern and classical techniques to determine the identity of an unknown aldehyde or ketone. Please follow this procedure:

1. Get unknown ^1H NMR spectrum via the class website. Get the unknown carbonyl compound from your TA. You should note its appearance (solid vs. liquid, color, etc.).
2. In the ^1H NMR spectrum, look for a peak between approximately 9–10 ppm. Note if you observe this peak, the ppm of the peak, and the appearance of the peak. A peak in this region usually corresponds to the H of an aldehyde, the RCHO hydrogen. Ketones will not have this peak in their ^1H NMR.
3. Make the semicarbazone derivative of your unknown according to the procedure on pg. 147–148 in the Lab Manual. You should dry your crystals by pressing them between filter paper before measuring the melting point.
4. Make the 2,4-dinitrophenylhydrazone derivative of your unknown. This reaction is described on pg. 198-199 and 206–207 of the Lab Manual. You will need to isolate the precipitate that results upon addition of 2,4-dinitrophenylhydrazine (2,4-DNP). Recrystallize the precipitate from a minimal amount of EtOH. Filter and dry the crystals before measuring their melting point.

For your lab report, you should draw the reactions for formation of the semicarbazone and for formation of the 2,4-dinitrophenylhydrazone from a generic aldehyde or ketone. You should include mechanisms for both these reactions.

Please note that these 2 derivatizations may not work well with all unknowns, but at least one should work.

Melting Points for Unknowns and their Derivatives

Unknowns	Aldehyde or Ketone	Melting Points (° C)	
		 semicarbazone	 2,4-dinitrophenylhydrazone
 isovaleraldehyde (3-methyl butanal)	Not reported	107	123
 2-pentanone	-78	112	143
 cyclopentanone	-51	210	146
 benzaldehyde	-26	222	237
 acetophenone	19–20	198	240
 acetone	-94	187	123