

EXPERIMENT 3

Nonaqueous Reactions of Metal Ions and Compounds

This experiment is assigned on page 95 of the textbook.

FOR THE INSTRUCTOR

This experiment can be performed in two ways.

1. The instructor demonstrates the reactions and the students, in groups, record the results and discuss their conclusions and record them in their lab reports (No worksheets have been provided).
2. The students perform the experiment in the laboratory, and the students, in groups, record the results and discuss their conclusions, and record them in their lab reports.

COMPOUNDS AND SOLUTIONS REQUIRED: Some of these may be left over from Experiment 2. The compounds should be anhydrous unless otherwise stated. Those indicated by # should be stored in an airtight manner, for example, in Erlenmeyer flasks with tightly-fitting rubber septa. In a glove bag we prepare individual portions of TiCl_4 in micro test tubes capped with septa.

$\text{BiCl}_3\#$	$\text{TiCl}_4\#$	CCl_4
$\text{SbCl}_3\#$	SnCl_4	HCl, concentrated
Acetone	Pyridine	Toluene, dry
HCl, 0.5 M	CsCl, 1 M (16.8 g/100 mL)	
$(\text{CH}_3)_4\text{NCl}$, saturated aqueous solution		

FOR THE STUDENTS

In this experiment, we see whether the reactivity of cations observed in Experiment 2 is still present in the absence of water, in nonaqueous solutions. We also try to identify products other than hydroxides or oxides that may form when cations react in the presence of water.

Part I

- A. 1. In a test tube, take 1 mL of 0.5 M HCl. Using first long, then short-range pH papers, measure the pH of this solution. Then in the hood add an eyedropper-full of pyridine (py), C_5H_5N (Fig. 3.4, p. 96), to the test tube. Stir with a glass rod, then measure the pH of the solution again.
2. What term do we use to describe what the pyridine has done to the HCl solution? What class of compound reacts with acids such as HCl in a manner similar to pyridine? Does it seem to be necessary for that class of compound to contain hydroxide or oxide ions? Try to write an equation for the above reaction, using full Lewis dot structures, to show how the pyridine reacts with the HCl to give the observed result.
3. In the hood, try holding the stopper from a bottle of concentrated HCl over a watch glass in which you have put an eyedropper-full of pyridine. Does it appear that this reaction can proceed even when water is not a factor? (Note: The water in the air and in the concentrated HCl are not involved in this reaction.)

Part II

- A. 1. Looking back at your results in Experiment 2, select the chlorides of three of the more acidic cations from that experiment (Suggestions: $TiCl_4$, $BiCl_3$, $SnCl_4$, and $SbCl_3$). If you select a liquid chloride, dissolve one-half of an eyedropperful in 5 mL of dry toluene in a test tube. If you select a solid chloride, dissolve two large spatula-tipfuls of it in 2–3 mL of acetone. To each test tube of a chloride that you prepare in this manner, add one-half of an eyedropperful of pyridine (in the hood). Describe the results.
2. By analogy with the reaction in **Part I** of this experiment, what is probably happening in this reaction? Draw Lewis dot structures of the products you think might have been produced in this reaction. The function of the pyridine in this reaction is to act as what class of compound? The function of the metal cation (or metal chloride) is to act as what kind of compound?

Part III

- A. 1. In each of three test tubes, take 3 mL of a 1 M aqueous solution of cesium chloride, CsCl. To each add two good spatula-scoopfuls or a half-eyedropperful of one of your chlorides from the previous part of this experiment. Stir. Let sit awhile if no reaction is immediately apparent.

2. Explain your results. Draw possible structures of the products. What is the function of the cesium chloride, and which ion (Cs^+ or Cl^-) is performing this function? (Note: Although water is present, it is not participating in this reaction either.)
3. If you are skeptical of that statement, as any good chemist ought to be, try the experiment with the three chlorides and 3 mL of water instead of the CsCl solution, and see if the results are quite the same.
4. Add a half-eyedropperful of CCl_4 to 3 mL of the 1 M CsCl solution. Explain the results (or lack of results) with CCl_4 .

Part IV

- A. 1. Repeat **Part III.A.1** using 3 mL of a saturated aqueous solution of tetramethylammonium chloride, $(\text{CH}_3)_4\text{N}^+\text{Cl}^-$, in place of the CsCl solution. Are your results similar?
2. Write a Lewis dot structure of a plausible product for one of the reactions. Which ion (chloride or tetramethylammonium) is directly involved? Compare or contrast the behavior of the two nitrogen-containing species, pyridine and the tetramethylammonium ion, using their Lewis dot structures to explain their similarity or dissimilarity.
3. In summary, what structural feature seems to be essential for a species to act as a base?