10.7 Lithium Chloride and Lithium Carbonate

10.7.1 Introduction

Because Lithium (like hydrogen and helium) does not give an EDS spectrum, other methods need to be utilized for identification. Lithium may be encountered as lithium chloride, lithium carbonate, lithium hydroxide, as a lithium-transition metal oxide in lithium batteries, or less commonly in a variety of other compounds. It is commonly encountered in the Birch reduction or "Nazi" synthesis of methamphetamine. Sodium metal (and sodium compounds, transition metal oxides, etc.) may also be used in this synthesis.

10.7.2 Instrumentation

- Fourier Transform Infrared Spectrometer (FTIR)
- Ion Chromatograph
- Crystal Tests & Chemical Spot Tests

10.7.3 Minimum Standards & Controls

Run Blanks and Controls for the procedure selected

See specific instrument's Minimum Standards & Controls

10.7.4 Procedure or Analysis

10.7.4.1

1) Run FTIR directly on the material.

10.7.4.2

2) Perform zinc uranyl acetate crystal test as follows:

10.7.4.2.1

a) Place sample on slide, dissolve in small drop of H2O

10.7.4.2.2

b) Place small drop of 10% acetic acid saturated with zinc acetate next to the other drop

10.7.4.2.3

c) Dissolve a few grains of uranyl acetate dihydrate in the 10% acetic acid/zinc acetate, mix well

10.7.4.2.4

d) Draw the 10% acetic acid/zinc uranyl acetate drop into the sample drop, mix briefly

10.7.4.2.5

e) Let stand a minute or so

10.7.4.2.6

f) Observe under microscope (400X) with cover slide

10.7.4.2.7

g) Note appearance of octahedra of the triple acetate, or what Chamot and Mason ("Handbook of Chemical Microscopy", Volume II, 2nd edition) call monoclinic polysynthetic twins (pseudo-rhombohedral), crystals. Run a sodium standard and lithium standard alongside the sample. Both sodium and lithium form these crystals. Run any other Group one (periodic table) standards you have available (potassium, cesium, etc.).

Run the test on a blank slide, as well.

10.7.4.3

3) Perform barium chloride test for carbonates, phosphates and sulfates. 5% BaCl2 added to a sample with either a phosphate, carbonate or sulfate will give a white precipitate. Addition of a drop of

concentrated acetic acid will dissolve a carbonate quickly, a phosphate slowly and a sulfate not at all. Run standards of all 3 salt types alongside the sample and observe under a stereomicroscope.

10.7.4.4

4) Perform silver nitrate test for the presence of chlorides (forms white precipitate if present).

10.7.4.5

5) Run SEM-EDS of material. If EDS spectra shows sodium, report out sodium, or identify salt form using ion chromatography, FTIR or both. If EDS shows no sodium, proceed as if lithium is present. Note other elements present that could account for the anion portion of the lithium compound(s) present. (i.e., nitrogen could mean ammonium salt is present), or the cation portion of any other inorganic compounds present.

10.7.4.6

6) Obtain acetone soluble portion (lithium chloride is soluble in acetone, most chloride salts are not). Run on FTIR to confirm LiCl, if present. Run FTIR of LiCl standard and chloride salts of other metals present on EDS spectra (i.e., AlCl3 and NiCl2) or obtain good library spectra for comparison purposes. Make LiCl identification after careful comparison with LiCl standard and other standard spectra. Infrared spectra of different chloride salts are very similar.

10.7.4.7

7) If LiCl was not identified in 10.7.4.6, obtain a methanol soluble portion of the acetone insoluble material from 10.7.4.6 (use excess MeOH). Dry MeOH soluble portion and check for lithium hydroxide on FTIR.

10.7.4.8

8) From the acetone insoluble and methonal insoluble material from 10.7.4.7, obtain an H2O soluble portion, collect and dry. Run sample on FTIR. Sample is probably not pure at this point.

10.7.4.9

9) Wash H20 soluble extract from 10.7.4.8 above with MeOH and CH2Cl2, and discard washes. Repeat if necessary.

10.7.4.10

10) Dry sample. Analyze on FTIR. If sample is lithium carbonate, run Li2CO3 standard through the same extraction procedure and analyze on FTIR. Obtain infrared spectra of other carbonate salts by either running standards or using library spectra. Compare to Li2CO3 if present.

FTIR spectra of carbonates vary a significant amount from cation to cation. (i.e. Li2CO3 has a different spectrum from that of Na2CO3)

10.7.4.11

11) If available, run sample on an ion chromatograph to determine, or verify the anion(s) present.

Note: Both lithium chloride and lithium carbonate are alkaline. Samples encountered may have a pH of 10-11. Some literature sources give lithium hydroxide as a product in the lithium reduction of ephedrine to methamphetamine. While it may occur in the equation, it may not be formed in all cases.

10.7.5 References

Chamot and Mason "Handbook of Chemical Microscopy", Volume II, 2nd edition