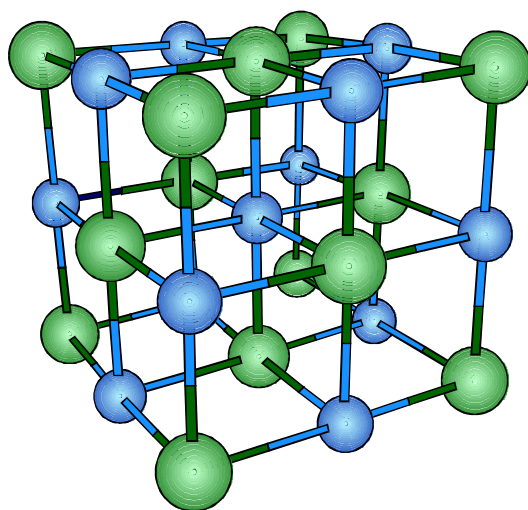


CHEMISTRY 323
Inorganic Chemistry

LABORATORY MANUAL



Department of Chemistry
Tulane University

Fall 1997

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SCHEDULE OF EXPERIMENTS

September 16-19	Experiment 1	Section 2
September 24-30	Experiment 1	Section 3 and 4
	Experiment 2	Section 2 and 3
October 1-7	Experiment 2	Section 4
	Experiment 3	Section 2
October 8-14	Experiment 3	Section 3 and 4
	Experiment 4	Section 3
October 15-21	Experiment 4	Section 2 and 5
October 22-28	Experiment 5	
October 29-November 4	Experiment 6	
November 5- 11	Experiment 6	
November 12-18	Experiment 7	
November 19-25	Experiment 7	
December 2-5	LAB FINAL	

Marking; All laboratory reports are due two weeks after the end of the experiment. Late reports will be penalized 5% per day except in extenuating circumstances. Each report will be worth 10% of your final mark, with the lowest grade being dropped in the final calculation. The final exam will be worth 20% of the grade.

UNDERGRADUATE TEACHING LABORATORY SAFETY REGULATIONS

1. NO STUDENT MAY WORK IN A LABORATORY UNLESS AN INSTRUCTOR IS ON DUTY.
2. RECOMMENDED EYE PROTECTION MUST BE WORN AT ALL TIMES IN THE LABORATORY, UNLESS SPECIFICALLY INSTRUCTED OTHERWISE.*
3. NO SMOKING, EATING OR DRINKING IS PERMITTED.
4. LABORATORY COATS ARE RECOMMENDED WHILE WORKING IN THE LABORATORY.

* **Regular prescription glasses are an adequate substitute only in the absence of an explosion hazard or where no hazardous chemicals are being used in the laboratory.**

In addition to these four basic rules, the following regulations should be observed:

1. No student may perform an unauthorized experiment.
2. Never leave an experiment in progress unattended.
3. Any chemical which produces toxic vapors must be used in a fumehood.
4. Wipe-up spilled chemicals and bottle `rings' immediately.
5. Never handle or pour flammable liquids near an open flame.
6. Report all accidents to the instructor immediately.
7. Unless given specific permission to the contrary, NEVER pipette a liquid by mouth; Use a rubber bulb.
8. Keep the sinks clean.
9. At the end of the period, make sure the hood, work area and sink are clean and tidy.

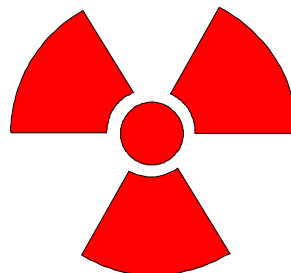
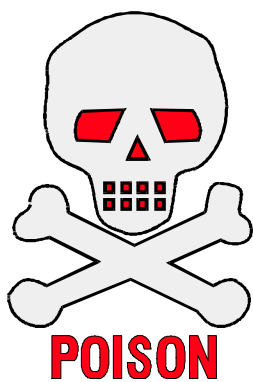
Suggestions

Learn the locations of the emergency shower, eyewash and fire extinguisher and know how to use them. While working in the laboratory, beware of burns from forgotten, still-lit burners and from hot glassware. Wash your hands at the end of each laboratory class.

If you are unsure about any directions, ask your instructor. For example, ask for his/her instructions when disposing of used chemicals. Finally, never hurry when performing experiments. Safety always has the highest priority.

WORKPLACE HAZARDOUS MATERIALS INFORMATION SYSTEMS (WHIMS)

WHIMS provides that, by law, students are entitled to information concerning any materials used in the laboratory. This material is available on a Material Safety Data Sheet (MSDS). These are available from Tulane's Office of Environmental Health and Safety.



CHEMISTRY

INORGANIC LABORATORY COURSE:

The experiments contained in this laboratory manual have been designed to contribute to the student's understanding of the principles of inorganic chemistry through:

- 1) The use of some of the modern techniques for the synthesis and investigation of inorganic compounds.
- and 2) The gaining of first hand experience of inorganic compounds. Note that the experiments are not in some cases specifically tied to particular sections of the lecture course, but rather designed to augment the lectures in order to provide a broad introductory course in inorganic chemistry.

LABORATORY REPORTS

The format of the written laboratory report will vary slightly from experiment to experiment, but in general they are expected to contain the following:

- (a) Purpose: Briefly describe what you plan to do, and why.
- (b) Procedure: Give a condensed version of what is in the manual. Include the exact weights of reagents/products you measured.
- (c) Observations: Describe what happened in your reaction, in some detail.
- (d) Analysis: If required, report the qualitative tests in the usual way (Test/Observation/Inference). Include any spectra you recorded and provide interpretations where possible.
- (e) Discussion: Give an explanation of the steps in the synthesis, and of the observations you made. Give balanced chemical equations wherever possible. Explain any anomalous (i.e. too low, or too high) yields from syntheses.
- (f) Conclusion: Give a very brief summary of your results. This will obviously differ greatly from experiment to experiment.
- (g) Post-lab questions Answer fully all questions posed at the end of the laboratory write-up.
- (h)References Include all references used in the laboratory write-up.

Laboratory reports should be written in third person. All reports must be typed or legibly handwritten. Graphical representation of data should be computer generated.

The laboratory reports will be graded on the basis of 100 points, distributed as follows.

(a) Purpose	10 pts
(b) Procedure	10 pts
(c) Observations and analysis	20 pts
(d) Discussion	20 pts
(e) Conclusion	10 pts
(f) Post Lab Questions	20 pts
(g) References	10 pts
<hr/>	
TOTAL	100 pts

SYNTHESIS OF A SOLID ACID, 12-Tungstosilicic acid, $\text{H}_4\text{SiW}_{12}\text{O}_{40}\cdot 7\text{H}_2\text{O}$

REFERENCES:

- a) E. North, *Inorg. Syntheses*, **1**, 129 (1939).
- b) N. N. Greenwood and A. Earnshaw, "Chemistry of the Elements" Pergamon Press, 1984, pp 1171-1186.
- c) F. A. Cotton and G. Wilkinson, *Advanced Inorganic Chemistry*, 5th Edition, pp 811-818.
- d) D. M. Adams and J. B. Raynor, *Advanced Practical Inorganic Chemistry*.

1. INTRODUCTION

The condensation of oxometalate anions in acidic solutions is a commonly encountered reaction in inorganic chemistry. For example, the following equilibria between molybdenum species are highly dependent on pH:



These ions are only two of the many complex species which occur in solution, and hydration, protonation and further condensation or hydrolysis reactions can increase the diversity of these systems. The basic building block of these isopolyanions is the MoO_6 octahedron, and these units can be connected by sharing corners, edges, but not faces. In some structures MoO_4 tetrahedra can also be found. Tungsten exhibits very similar chemistry in this regard.

There has been renewed interest in these types of compounds, largely as a result of their potential and actual, use as catalysts. They have found use in selective oxidation and acid catalyzed reactions.

In this laboratory exercise you will prepare a heteropolymetalate species which is a solid acid. The object of the experiment is to:

- a) Prepare the compound 12-tungstosilicic acid using a solvent extraction method.
- b) Quantitatively determine the available protons in this material.
- c) To test the material as a solid acid catalyst.

2. PROCEDURE

This entire procedure should be carried out in the fumehood. Ensure that there is no naked flame in the hood or close to you.

Dissolve 15 g of sodium tungstate dihydrate, $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$, in 30 ml of water and add 1.16 g of sodium silicate solution (density 1.38 g/cm^3). Stir the solution vigorously at just below the boiling point, and add concentrated hydrochloric acid (10 mL) dropwise over a period of about 30 minutes, using a dropping funnel. Cool the solution to room temperature, then filter it, add a further 5 mL of concentrated hydrochloric acid slowly, and transfer it into a separatory funnel. Shake the solution with diethyl ether (12 mL); at this point you should observe three layers in the funnel. If not, add a little more ether, shake and again allow to separate. Withdraw the bottom, oily ether layer and save it in a beaker. Repeat the extraction process several times, until the yellow product in the middle layer has been completely removed. Discard the liquid left in the funnel (put it in the residues bottle), rinse out the funnel, and return the ether extracts to the funnel, together with a solution of 4 mL of concentrated hydrochloric acid in 12 mL of water, and an additional 4 mL of ether. After shaking, run off the lower (ether) layer into an evaporating dish and allow the solvent to evaporate. Dry the white crystalline product at 70°C for about two hours, then put it into a tared sample vial, reweigh the vial and record your yield.

DO NOT USE A METAL SPATULA TO HANDLE YOUR PRODUCT, OR IT WILL TURN BLUE!

3. Determination of the acidity of the product

Weigh out about four grams of the product and dissolve it in deionized water, and dilute to 100 mL in a volumetric flask. Titrate 40 mL aliquots of the solution with the 0.1 M NaOH solution provided, using an appropriate indicator (e.g. methyl orange or chlorophenol red). Assuming the formula given in the title, calculate the moles of titratable protons per mole of compound.

4. Test of $\text{H}_4\text{SiW}_{12}\text{O}_{40} \cdot \text{XH}_2\text{O}$ as a Solid Acid

Add 6 drops of cyclohexanol to 3 mL of cyclohexane. Shake to dissolve. Add ca. 0.2 g of $\text{H}_4\text{SiW}_{12}\text{O}_{40}$ and shake for a few minutes. Filter to remove the solid acid from the solution A. Prepare a second solution B of 6 drops of cyclohexanol in 3 mL of cyclohexane. Add ca. 10 drops of dilute bromine water (pale brown color) - 20 drops of saturated Br_2 water in 20 mL of distilled water - to each of the cyclohexane solutions and shake. Solution A should decolor, and a white precipitate of $\text{C}_6\text{H}_{10}\text{Br}_2$ may appear in the organic layer.

Solution B will extract the brown Br_2 out of the Br_2 water into the organic layer, but the organic layer will not decolor.



NB: It is important to filter out the solid acid after reaction with cyclohexanol since it appears to react with bromine water by itself.

Post-lab questions

1. What is a Keggin unit? Without giving a complex diagram briefly describe what is meant by this term.
2. Give two examples, other than that made in this experiment, of compounds which are "solid acids" and which can be used as acid catalysts.
3. What other atoms can occupy the tetrahedral position in the center of the M_{12} polyanion in which the Si is found in $H_4SiW_{12}O_{40} \cdot 7H_2O$?

TIN(IV) HALIDES

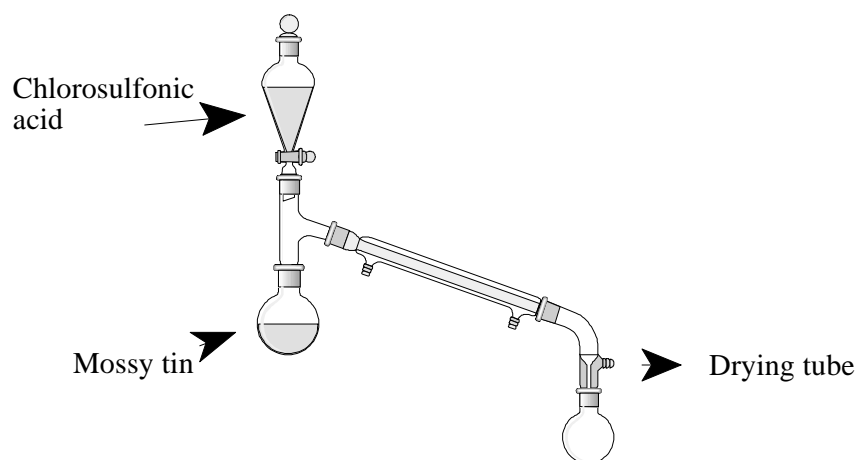
1. Introduction

Read the appropriate section of F. A. Cotton and G. Wilkinson, *Advanced Inorganic Chemistry*, 5th Edition in order to acquire some background for interpretation of the observations in Part 4 of this experiment.

2. Preparation of Tin(IV) Chloride

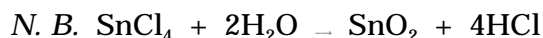
 **[CAUTION: ClSO_3H REACTS VIOLENTLY WITH WATER.]**

Because the handling of reagents used in this experiment could be hazardous, *consult your instructor* before beginning. You **MUST** use a fumehood, and keep the window between you and the experiment. It is important that the apparatus you use is properly dried before you start.



Apparatus for the Synthesis of Tin(IV) Chloride

Put 5.5 g of mossy tin into a 25 ml semi-micro distillation flask and slowly add 13 ml of ClSO_3H (chlorosulfonic acid) from a tap funnel. The addition should be just fast enough to maintain a steady reaction, and to provide enough heat to distil the stannic chloride into the receiving flask. When the reaction is complete, redistill the product, if necessary. It should be a colorless liquid, b. p. 110°C .



As soon as the product is obtained, stopper it in a flask.

3. Preparation of Tin(IV) Iodide

Weigh 1.0 g of granulated tin and 3.3 g of iodine into a 25 ml flask. Add 15 ml of carbon tetrachloride and one or two small boiling chips and fit a reflux condenser to the flask. Warm the flask gently, using a water bath on a hot plate, until the reaction starts and then remove the source of heat.

Re-apply heat as necessary to maintain a steady refluxing of the solvent until no free iodine remains. (Solution is orange-red instead of violet.)

Remove the condenser, heat the solution to boiling point and filter through a fluted filter paper to remove excess tin. The filtering equipment should be preheated with hot solvent in order to prevent the product from crystallizing prematurely. Wash the residue in the flask and on the filter with 3 ml of hot carbon tetrachloride, combining the washings with the main filtrate.

Cool the solution in an ice bath and filter off the solid. Evaporate the filtrate to about half-bulk to obtain a further crop of crystals. Weigh and record the yield. Calculate the theoretical yield (based on iodine).

The solid may be recrystallized from carbon tetrachloride.

4. Reactions of Tin(IV) Halides

Compare reactions of the two halides in the following tests explaining, as far as possible, the differences and similarities.

(a) Solubility in (i) ethanol, (ii) benzene, (iii) dilute hydrochloric acid, (iv) concentrated

- hydrochloric acid, (v) 2M-sodium hydroxide solution. Note color of solutions, possible reactions, and write equations to explain all observations.
- (b) Stability to oxidation, *e.g.* acidic permanganate solution.
 - (c) Stability to reduction, *e.g.* acidic stannous chloride, zinc metal and dilute hydrochloric acid.
 - (d) Complexation, or "adduct"-forming reactions. To a solution of 2.0 g triphenyl phosphine in 2 ml benzene, add a solution of 0.5 g of SnCl_4 in 2 ml benzene. Repeat this reaction with 0.5 g SnI_4 . How does the reactivity of SnCl_4 change as a result of forming a complex with Ph_3P ?
 - (e) Although SnCl_4 will react with triphenylphosphine, CCl_4 is unreactive in this regard. Explain why this is so.

Post-lab Questions

1. Based on the solubility properties of the tin(IV) halides, how do they differ from the more commonly encountered metal halides of metals such as sodium or magnesium?
2. What is an "adduct" ? Identify an example from the chemistry of SnCl_4 , and also one from the chemistry of boron.
3. What structures would be predicted for SnCl_2 , SnCl_4 , and $\text{Cl}_4\text{Sn} \cdot \text{PPh}_3$ using VSEPR theory. (Assume SnCl_2 is monomeric)

REDOX CHEMISTRY OF CHROMIUM

1. Preliminary Exercises

Consult F. A. Cotton and G. Wilkinson, *Advanced Inorganic Chemistry*, 5th Edition for a list of the oxidation states of chromium.

2. Reactions with Hydrogen Peroxide

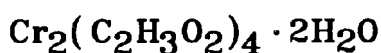
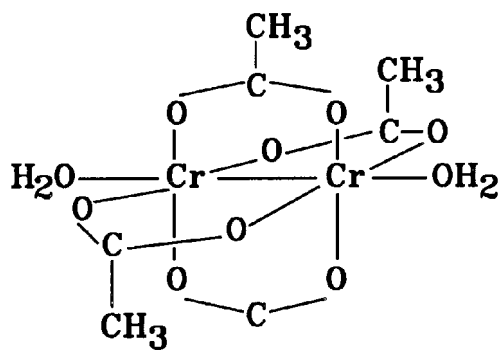
Carry out the following tests. Record your observations and explain them on the basis of the information gleaned from the preliminary exercises.

- (a) Dissolve 25 mg of chromium(III) sulfate in 2 ml of water. Divide the solution into 2 parts. To one add 1 ml 2M-sulfuric acid, to the other add 1 ml 2M sodium hydroxide solution. Then to each add 1 ml hydrogen peroxide (6% solution). After observing any initial reaction warm the solution.
- (b) Dissolve 25 mg of potassium chromate in 2 ml of water. Divide the solution into 2 parts and repeat the test described in (a).

3. Preparation of Chromium(II) Acetate

Chromous acetate as a dimeric, relatively insoluble material is a relatively stable chromium(II) compound, the rate of oxidation being sufficiently slow to enable it to be prepared without the elaborate precautions of oxygen, which other chromous preparations demand.

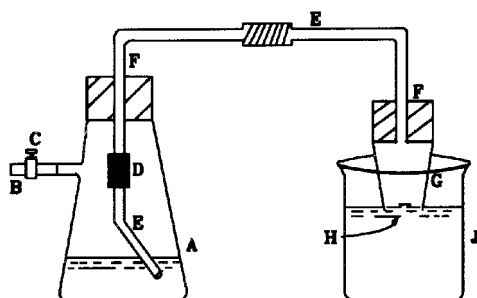
Each chromium ion in the dimer has six octahedral bonds, four to the oxygen atoms of the acetate groups (which act as bridging ligands between the two chromium ions), one to a water molecule and the sixth a mutual chromium-chromium bond.



The acetate is of interest in that it is diamagnetic. The 3d electrons have 'paired-up' in forming the dimer (evidence for a weak chromium-chromium bond).

Weigh 10 g of potassium dichromate into a 250 ml beaker and add 50 ml of concentrated hydrochloric acid. Place the beaker near a fume extraction vent and simmer gently for about 30 minutes. Allow to cool.

During this time assemble the apparatus shown in the diagram below:



- A = filter flask (500 ml)
- B, D = rubber tubing connections
- C = screw clip
- E = glass tubing (3 sections)
- F = rubber bungs
- G = Gooch crucible
- H = glass wool pad
- J = beaker (600 ml)

Weigh 30g of mossy Zn into flask A. Weigh 50g of sodium acetate into 600 ml beaker J, then add 150 ml of water. Cool the solution and add a few pieces of dry ice to remove oxygen, then warm gently to dissolve the salt.

Add a similar quantity of carbon dioxide to a 400 ml beaker containing 300 ml of water. Cover this beaker with a watchglass, and add more carbon dioxide as required to maintain an

inert atmosphere over the water. This is to provide oxygen-free water to wash the chromous acetate.

Transfer the prepared chromic chloride solution to flask A. Ensure that clip C is open. Add* 50 ml of concentrated hydrochloric acid to A and fit the bung carrying tube E firmly into place. Place beaker J containing sodium acetate solution with the end of the Gooch crucible just immersed in the solution.

Allow the reaction in A to continue until the solution has a clear sky-blue color. Then close C so that the solution is forced through E and H into J. The chromous acetate precipitates immediately as a red-brown solid.

The product needs to be protected from oxygen while you are working it up. A simple way is to add a few small pieces of dry ice to the Büchner funnel in which you are filtering off the precipitate. This will generate an atmosphere of CO_2 over the solution, and the sample. After filtering off the solid wash it with oxygen free water, then finally with acetone. Dry the product in a vacuum desiccator.

5. Analysis of the Chromium(II) Compound

The chromium content is determined by conversion to CrO_4^{2-} , and the measurement of the intensity of the yellow color using the spectrophotometer.

A weighed sample of the compound** is dissolved in dilute nitric acid to give a solution of various Cr^{III} ions. Make the solution up to 100 ml in a volumetric flask, take a 5.0 ml aliquot and place it in a 250 ml Erlenmeyer flask. Add enough 2.0 M NaOH to neutralize the free acid, and then add 10.0 ml more NaOH. Add about 5-10 drops of 30% hydrogen peroxide, then heat on a steambath for a few minutes until oxygen evolution ceases. Cool to room temperature and make up to 250 ml. A duplicate determination should be made, and also in this case a blank solution (*same reagents and procedure but no Cr*).

Measure the absorbance of both solutions and blank in a 1 cm path length cell at 374 nm and calculate the concentration of chromate ion from the relationship:

* The reaction of conc. HCl with zinc can be quite violent. It would seem wise to add the acid portionwise to the Cr(III) chloride solution in the reaction flask, and to mix it by swirling to avoid the concentrated acid forming a dense lower layer in contact with the zinc, without dilution.

** Calculate the weight required by assuming $n = 1$, $A = 0.5$ in the formula below.

$$= \frac{A}{C}$$

= molar extinction coefficient = $4820 \text{ L mol}^{-1} \text{ cm}^{-1}$ at 374 nm

A = absorbance - measured by experiment

c = molar concentration - calculated from measured value of A

= path length of cell - 1 cm

Remember the dilution step when calculating chromium content of the original sample.

Post-lab Questions

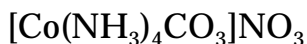
1. Draw the structure of the chromium(II) acetate molecule, pointing out with notes any special features it might have.
2. Write a balanced chemical equation for the oxidation of Cr(III) to chromate(VI) by hydrogen peroxide in alkaline solution.
3. Calculate the Cr content (mass %) of a sample of a complex given the following: 0.10 g of the sample was dissolved in dilute nitric acid, and made up to 100 mL. 50 mL of this solution was taken, and the Cr(III) converted to chromate by the addition of sodium hydroxide and hydrogen peroxide. The solution was made up to 250 mL and its absorbance was measured at 374 nm in a 1 cm pathlength cell. $A = 0.5$; $\epsilon = 4820 \text{ L mol}^{-1} \text{ cm}^{-1}$.

COORDINATION COMPLEXES - COBALT(III) AMINE COMPLEXES

1. Preliminary Exercises

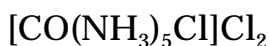
- (a) Read the appropriate pages of, for example, Cotton and Wilkinson on cobalt(II) and cobalt(III) complexes.
- (b) The book *Synthesis and Technique in Inorganic Chemistry* by R. J. Angelici has some useful information on this experiment.

2. Preparation of Tetraaminecarbonatocobalt(III) Nitrate



Dissolve 20 g of ammonium carbonate in 60 ml of water and add 60 ml concentrated aqueous NH_3 (solution A). Dissolve 15 g of cobalt(II) nitrate hexahydrate in 30 ml of water (solution B). Mix solution A with solution B and add 8.0 ml of 30% hydrogen peroxide solution (slowly), stirring continuously. Pour this solution into an evaporating dish and concentrate to about 90 ml on a hot plate. During the evaporation add in small portions, a total of 5 g of ammonium carbonate. Filter the hot solution and cool the filtrate in an ice-water bath. When crystallization is complete, filter off the red crystals under suction, and wash with 2-3 ml of water then two or three times with 10 ml of ethanol. Preserve some sample for conductance measurements and use 5 g for the next preparation.

3. Preparation of Pentaaminechlorocobalt(III) Chloride



Dissolve 5 g of the prepared tetraaminecarbonato complex in 50 ml of water, and add 10 ml of concentrated hydrochloric acid. Neutralize the solution with concentrated aqueous ammonia, and add 5 ml in excess. Heat in an evaporating dish on the steam bath for 20 mins., and then cool slightly and add 75 ml of conc. HCl. Re-heat on the steam bath for a further 10 mins. and then cool. Filter and wash the purple crystals with 5 ml of ice-cold water, (*on a Büchner funnel*), then with two portions of 15 ml of ethanol, followed by two 15 ml portions of acetone. Allow to dry and record the yield. Calculate a theoretical yield and a percentage yield for **each** of the cobalt(III) complexes.

4. Measurement of Electrical Conductance

The number of ions constituting a given compound can, in favorable cases, be determined by measuring the electrical conductance in solution.

A number of definitions are necessary:

Specific resistance ρ = the resistance in ohms of a solution in a cell which has two 1 cm² electrodes separated by 1 cm.

$$\text{Specific conductance } L = \frac{1}{\rho}$$

Resistance of a non-standard cell, $R = k\rho$ (k = conversion factor the cell constant)

$$\text{Thus } R = \frac{k}{L}$$

Thus, one first calibrates a cell using a standard solution of electrolyte. Using a solution whose specific conductance (L) is known enables the constant k to be determined. Then knowing k , the value of R for any solution will enable L to be calculated. The **Molar Conductance** Λ can then be calculated.

$$\text{Molar Conductance } \Lambda = \frac{1000 L}{M} \quad (\text{M} = \text{Molarity of solution})$$

For some known cases, typical values of Λ are:

Number of Ions	Λ
2	118 - 130
3	235 - 275
4	408 - 435
5	550

5. Experimental Procedure

Consult your Demonstrator before using the Conductance Bridge.

- (a) Prepare a 0.0200 M solution of KCl in distilled water and use it to calculate the cell constant k . Specific conductance at 25° C is $2.768 \times 10^{-3} \text{ ohm}^{-1}$.

- (b) Prepare 0.001 M solutions of (A) and (B) and measure the conductance of these. Calculate Λ values and compare them with the above. Be sure to rinse the cell with distilled water before and after use.

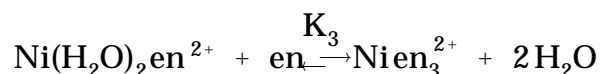
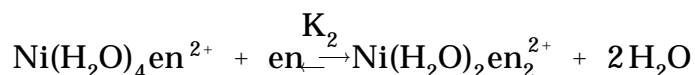
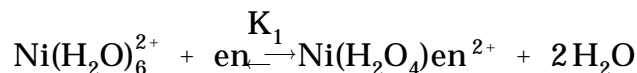
Post-lab Questions

1. Draw the structure of the tetraaminecarbonatocobalt(III) cation.
2. What can you determine about the structure of a coordination complex by measuring its conductance in a solution of known concentration?
3. Predict how many ions would be produced per formula unit of the compound $\text{Co}(\text{NH}_3)_4(\text{NO}_2)_3$, assuming it is monomeric, and contains an octahedrally coordinated cobalt?
4. Write a balanced equation for the oxidation of Co(II) to Co(III) by hydrogen peroxide. Why is this oxidation process assisted by the presence of ammonia?
5. Nitrate and carbonate are isoelectronic and isostructural anions. How could you try to show that the real formulation of tetraaminecarbonatocobalt(III) nitrate is as written, rather than its ionization isomer?

COMPLEX ION COMPOSITION BY JOB'S METHOD

1. Introduction

The nickel(II) ion in aqueous solution forms complexes with 1,2-diaminoethane(ethylenediamine) whose ligand replaces two water molecules in the hydrated complex, i.e. acts as a chelate.



The relative values of k_1 , k_2 and k_3 will determine the relative proportions of the complexes. If $k_3 \gg k_2 \gg k_1$, then addition of ligand will result in formation of Ni en_3^{2+} . If $k_3 \ll k_2 \ll k_1$, then all four complexes (or three, depending on the absolute magnitude of the constant) will co-exist in appreciable quantities at equilibrium. The application of Job's Method is limited to situations where one stage predominates.

In the method, the total molar concentration of nickel(II) plus ligand is kept constant and the ratio varied from zero to infinity. Initially in a solution containing no nickel(II) ion (ratio = 0), no complex will be present and the absorbance of the solution is due solely to diaminoethane. As the concentration of nickel(II) is increased, absorbance will increase (*due to formation of complex*) until the molar ratio of nickel(II) to ligand equals the ratio in the complex. Under the Job conditions, i.e. total molar concentrations of the two species is constant, this represents the maximum possible concentration of complex. Further increases in nickel(II) concentration (with corresponding decreases in ligand concentration) will cause

a decrease in absorbance until a solution containing no ligand (ratio =) has an absorbance due to nickel(II) ions alone. A plot of absorbance against ratio of molar concentration of nickel(II) ion to ligand will show a maximum where the ratio represents the composition of the complex.

Reference

The Job Method outlined above can be justified rigorously, see *Synthesis and Technique in Inorganic Chemistry* by R. J. Angelici.

2. Procedure

Using solutions of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ (0.4 M) and ethylenediamine (0.4 M) prepare solutions having a total volume of 10 ml in which the mole fraction of ethylenediamine, X, is 0.0, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8 and 0.9. Determine the absorbance of each solution at the following wavelengths: 530, 545, 578, 622 and 640 μm .

By appropriate graphical methods, obtain a possible formula to represent the predominant species present in nickel(II)-1,2-diaminoethane solutions.

Post-lab Questions

1. Write structural formulas for the three possible products of reaction of ethylenediamine with $\text{Ni}(\text{H}_2\text{O})_6^{2+}$.
2. What does the absorption spectrum of the ligand, ethylenediamine, look like in the visible region which you used in this experiment. Assign the absorption maxima you used in this experiment to individual species in the system, where this is possible.
3. What would happen if the values of K_2 and K_3 were larger than K_1 ? Could you still use Job's method?
4. From the observed changes in the spectra and a knowledge of the complexes present in the solutions, what can be said about the relative ligand field strengths of en and H_2O ?

INFLUENCE OF LIGAND FIELD TETRAGONALITY ON THE GROUND STATE SPIN OF NICKEL(II) COMPLEXES

David M. L. Goodgame

Special Safety Precautions

N,N-Diethylethylenediamine should be stored and used in a fume cupboard and any skin contact with it avoided; wear rubber gloves when using it. Also wear rubber gloves when working with solutions of the nickel salts and their complexes, to avoid the possibility of heavy metal skin allergy.

Regularly octahedral, first series transition-metal complexes of electronic configuration d^n ($n = 4-7$) may be either high- or low-spin depending, primarily, on the strength, or $10 Dq$, of the ligand field. For distorted ligand fields, a wider range of possibilities exists for changes in the ground-state spin. Changes in spin can have important effects, e.g. high-spin octahedral iron(II) complexes are much more labile than their low-spin counterparts. Moreover, the heme complexes involved in hemoglobin, catalase, cytochrome C, etc., contain the iron atom in either a high- or low-spin state, depending on the axial ligand.

The effects of changes in the axial ligands, X , on the electronic properties of a tetragonally distorted complex $trans\text{-ML}_4X_2$ are well illustrated by the magnetic properties and $d-d$ spectra of a series of nickel(II) complexes of formula $Ni(\text{Et}_2\text{en})_2X_2$, where $\text{Et}_2\text{en} = N,N$ -diethylethylenediamine ($\text{Et}_2\text{NCH}_2\text{CH}_2\text{NH}_2$) and $X = \text{Cl}^-, \text{Br}^-, \text{I}^-, \text{NCS}^-$, etc.

In the following experiment, a number of compounds of this type can be made and their

spin-states determined by magnetic susceptibility measurements, supplemented, if possible, by their solid state (reflectance) *d-d* spectra.

Experimental

a) $\text{Ni}(\text{Et}_2\text{en})_2(\text{NCS})_2$

Prepare an ethanol solution of nickel thiocyanate by dissolving nickel nitrate hexahydrate (2.0 g) and finely powdered potassium thiocyanate (1.3 g) separately in hot absolute ethanol and mixing the two solutions. Keep the volume of solution to a minimum and cool the mixture thoroughly before filtering off the precipitated potassium nitrate. (For your guidance, the solubility of nickel nitrate in hot ethanol is *ca.* 50 g/100 ml, and that of *powdered* potassium thiocyanate is *ca.* 10 g/100 ml).

To the resulting solution of nickel thiocyanate, add 2 ml of Et_2en by means of a syringe, and stir or shake the solution during the addition. Filter off the precipitated complex, wash it with a little cold ethanol, and dry it (air dry or vacuum desiccator). Record the yield of crude product and then recrystallise it from methanol. Record the yield of pure, crystalline product and take its m.p.

b) $\text{Ni}(\text{Et}_2\text{en})_2\text{I}_2$

Prepare an ethanol solution of nickel iodide containing *ca.* 2 g NiI_2 from ethanol solutions of the appropriate amounts of nickel nitrate hexahydrate and sodium iodide (solubility of NaI in hot ethanol *ca.* 16 g/100 ml). (Again, cool the solution well before filtering off the precipitated sodium nitrate). Add Et_2en (2 ml) using a syringe and filter off, wash (ethanol) and dry the precipitated complex. Do not attempt to recrystallise it. (Alternatively, or additionally, the analogous nickel nitrate complex can be made by simple addition of Et_2en to a concentrated ethanol solution of nickel nitrate).

b) Physical measurements

Measure the magnetic susceptibilities of the complexes and, in the case of the thiocyanate complex, also calculate the magnetic moment. If the thiocyanate complex has formed large crystals, it may be necessary to powder some of the sample to achieve good packing in the Gouy (or Evans) tube used for the magnetic measurements.

Obtain the IR spectrum of the thiocyanate complex and identify the bands due to the NCS

group, which coordinates to the nickel via the nitrogen atom, i. e. it is an isothiocyanato complex. If you have access to a spectrometer capable of measuring the reflectance spectra of solid samples in the UV-vis region, record the spectra of the complexes and relate the spectra to the colors of the compounds.

c) Other complexes

The analogous complexes with nickel chloride and nickel bromide exist as dihydrates as well as anhydrous compounds. Investigate the formation of these compounds by dissolving 1 g of nickel chloride or bromide respectively, in ethanol and adding Et₂en (1 ml) to each of these solutions. Observe the colours of the initial precipitates and any colour changes that take place when the products are filtered off, washed with ethanol and air dried. Put a small amount of each compound in a melting-point tube, record the melting point or decomposition point and note any colour changes that occur prior to that point.

If you have isolated both anhydrous and hydrated forms of each complex, record their infrared spectra (using *dry* Nujol) and identify additional bands from the water molecules. If time permits, the weight loss when one of the dihydrates is converted to its anhydrous analogue can be determined, for example, by heating in an oven or a drying pistol at ca. 100 °C, or on prolonged storage in an evacuated desiccator over a good drying agent.

Exercises

Draw the crystal field splitting diagram expected for a tetragonally elongated octahedron and use it to explain why some of the compounds you have made are diamagnetic and the others are paramagnetic with magnetic moments indicative of two unpaired electrons per nickel atom.

Structural, Electrical and Magnetic Properties of Perovskite Ceramics

Colin Greaves

Special Safety Precautions

Barium salts are very toxic. Due to the involatile nature of the barium compounds studied in this experiment, the use of a fume cupboard is unnecessary, but care should be taken to avoid ingestion during all handling operations.

The perovskite structure (Fig. 1), which is adopted by many oxides with formula ABO_3 , is very versatile, and many perovskites have useful technological applications (*e. g.* as ferroelectrics, catalysts, sensors and superconductors). In this structure, the A and O ions together form a cubic close-packed array, and the B ions occupy 1/4 of the octahedral holes. This experiment involves the synthesis of four compounds which are structurally closely related to perovskite, but have very different physical properties. Control of the types of cations in the large sites (A) allows some variation of the oxidation state of the smaller octahedral cations (B), and this confers the variable physical characteristics.

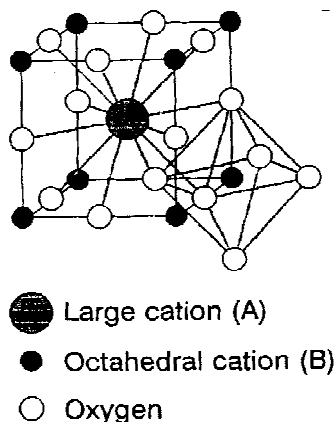


Figure 1. The perovskite structure adopted by many ABO_3 compounds.

Transition-metal ions with unpaired electrons are paramagnetic, provided interactions between neighbouring ions are weak; this "magnetically dilute" situation occurs in solutions and many solids. The perovskite structure allows quite strong interactions to occur *via* covalence in the M - O - M bonds, and this may result in ordering of the magnetic moments to give "ferromagnetic" or "antiferromagnetic" materials. At elevated temperatures, both classes are paramagnetic (random arrangement of magnetic moments), but below a critical temperature, the moments order in a parallel (ferromagnetic) or anti-parallel (antiferromagnetic) fashion. This temperature is known as the Curie temperature (T_C) for ferromagnets and the Néel temperature (T_N) for antiferromagnets.

The synthesis of solid state ceramic samples may be achieved by a variety of techniques. The simplest exploits the finite ionic or atomic diffusion processes which occur in mechanically mixed reactants at elevated temperatures and, in general, this allows precise control of the product's stoichiometry. An alternative approach involves the precipitation of a precursor, which can easily be converted to the final product by heating. This method assures intimate cation mixing prior to heating such that lower temperature may often be used for the final heating stage. In this experiment, both methods are used.

CaMnO_3 , $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$ and $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ are prepared from precipitated precursors, whereas $\text{YBa}_2\text{Cu}_3\text{O}_7$ is prepared from a mechanical mixture of Y_2O_3 , BaCO_3 and CuO . For $\text{YBa}_2\text{Cu}_3\text{O}_7$, it is not easy to ensure the correct cation ratio by simple precipitation methods, and a ceramic grinding and sintering technique is preferred. CaMnO_3 is a paramagnetic insulator at room temperature, whereas $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$ is paramagnetic but electrically conducting, and $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ is both electrically conducting and ferromagnetic. $\text{YBa}_2\text{Cu}_3\text{O}_7$ is metallic at room temperature, but becomes a superconductor with zero resistance to d. c. currents below 93K. Superconductors are perfectly diamagnetic, and it is this property which is examined in this experiment.

Experimental

a) $\text{YBa}_2\text{Cu}_3\text{O}_7$

If it is preferable to use reagents (Y_2O_3 , CuO , BaCO_3) which have been dried (e.g. 2 hours at 400 °C in a muffle furnace). Accurately weigh out about 0.5 g of BaCO_3 and the corresponding amounts of Y_2O_3 and CuO to give a Y :Ba :Cu ratio of 1:2:3 (0.1430 g and 0.3023 g respectively

for 0.5000 g of BaCO_3). Grind the materials together in a clean pestle and mortar until no white streaks are observed on grinding (ca. 10 min). Press 1 or 2 pellets (1-2 mm thick, 13 mm diameter) of the mixture at ca. 5000 kg and place the pellets in an alumina boat. Using a furnace with a programmable controller, subject the pellets to the following thermal program in air:

- 1) Heat to 930 °C and hold for 12 h
- 2) Cool to 500 °C and hold for 1 h
- 3) Cool to 400 °C at 50 °C h⁻¹
- 4) Cool to room temperature

When the furnace temperature is below 400 °C, the samples may be removed using tongs and placed on an insulating board until cold.

b) CaMnO_3

Dissolve 2.36 g of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and 2.87 g of $\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in 50 cm³ of distilled water. Whilst stirring the solution (magnetic stirrer), slowly add 100 cm³ of 1 M KOH using a separating funnel (about 3 min). After standing for 15 minutes, the brown precipitate should be filtered using a large Buchner funnel and washed thoroughly with distilled water (to remove excess KOH and KNO_3). Pump the sample as dry as possible, transfer to a watch glass and dry at 200 °C in a drying oven (ca. 1 h). Grind the sample and press two pellets 1-2 mm thick. The pellets should be placed in a porcelain or alumina boat and heated at 1000 °C for about 12 h. When the furnace has cooled to below 400 °C, the boat may be removed using tongs and placed on an insulating board until cold.

c) $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$ and $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$

Repeat the procedure described for CaMnO_3 , but use the following reagents:

$\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$: 3.68 g of $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$; 0.32 g of $\text{Sr}(\text{NO}_3)_2$; 2.87 g of $\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$;
 $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$: 3.03 g of $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$; 0.63 g of $\text{Sr}(\text{NO}_3)_2$; 2.87 g of $\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$.

d) Physical Properties

Place one of the pellets of each oxide in turn on a piece of paper and note its behaviour

when a bar magnet is placed under the paper. Cool the pellet of $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$ in liquid nitrogen using nylon forceps and re-examine its response to the magnet. If necessary, the pellets may be ground in order to examine the magnetic properties. Grind one of the pellets of CaMnO_3

using a clean pestle and mortar. Determine the magnetic susceptibility and effective magnetic moment of Mn^{4+} using any suitable method (*e. g.* a Johnson Matthey magnetic balance).

Cool one of the pellets of $\text{YBa}_2\text{Cu}_3\text{O}_7$ in liquid nitrogen, and quickly place a small magnet (Nd-Fe-B or Sm-Co) above it. The diamagnetic properties of a superconductor should allow you to float the magnet above the sample.

For each sample, measure the electrical resistance of one of the pellets using a suitable method (qualitatively, differences should be observable using a simple DVM in resistance mode, but quantitative measurements will require the use of a conventional 4-probe dc method, if available).

CaMnO_3 gives an X-ray powder diffraction trace with the first six reflections at 2θ values of 23.85° , 34.00° , 41.93° , 48.83° , 55.06° and 60.82° (wavelength 1.542 \AA). If X-ray diffraction facilities are available, the pattern can be recorded for the CaMnO_3 sample prepared. Confirm the primitive cubic structure of perovskite and determine the unit cell size.

Exercises

The structures of all four compounds are related to perovskite (Fig. 1). If available, a model of the structure should be examined. There are two independent cation arrays: the first consists of octahedrally coordinated ions, which are Ti^{4+} in the parent CaTiO_3 and Mn/Cu ions in the compounds synthesised; the second array has 12-coordinate ions which are Ca^{2+} in CaTiO_3 , La/Sr in $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$, etc. In fact, the Mn perovskites prepared all show minor deviations from the ideal cubic structure, due to size and electronic effects.

For an ideal, undistorted perovskite ABO_3 in which each cation (ionic radii r_A and r_B) contacts the coordinating O^{2-} ions (radius r_O), show that

$$r_A + r_B = \sqrt{2}(r_B + r_O)$$

Generally, some tolerance is allowed such that

$$r_A + r_B = t\sqrt{2}(r_B + r_O)$$

where the tolerance factor t is 0.8-1.0. For undistorted perovskites, t is high, e.g. 0.99 for SrTiO_3 . From a table of ionic radii, determine t for CaMnO_3 .

Mn^{3+} has the electron configuration $t_{2g}^3e_g^1$ and is therefore likely to show a substantial JahnTeller distortion. In fact, the distortion is cooperative as shown in Figure 2. In the layer shown, for example, each Mn has 2 short Mn - O bonds and 2 long bonds. If the O ions above and below the Mn ions have short bonds, all the Mn ions can achieve a similar distorted stereochemistry. Explain why the distortion shown in Figure 4.23-2 should stabilise the structure.

The electrical and magnetic properties of Mn perovskites are critically dependent on the Mn oxidation state. What is the formal oxidation state of Mn in CaMnO_3 , $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$, and $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$? The conductivity of many transition-metal oxides may be related to a simple mechanism involving the hopping of electrons between two transition-metal ions. Using such a mechanism and considering the energy involved when an electron hops from one Mn ion to a neighbouring Mn ion, explain the difference in conductivity between CaMnO_3 , and $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$ and $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$. In the determination of the effective magnetic moment, μ_{eff} , for Mn^{4+} , the Curie Law is used:

$$\chi_a = \mu^2/8T$$

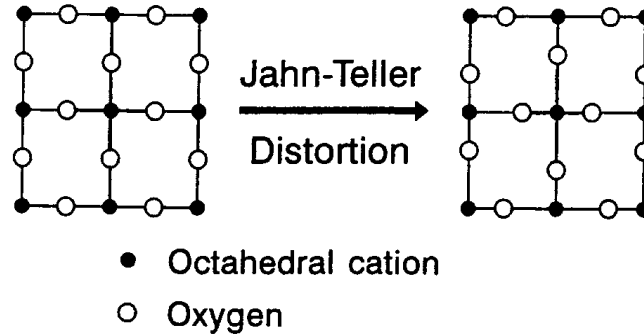


Figure 4.23-2. Cooperative Jahn-Teller distortions in a layer of the perovskite structure.

Compare your value μ_{eff} with μ_{so} , the spin-only moment, which is the magnetic moment expected if only electron spins contribute to μ_{eff} . The main reason for the disagreement is that CaMnO_3 is antiferromagnetic at low temperatures (T_N ca. 120 K). Interactions between magnetic moments are still apparent at higher temperatures and result in an apparent reduction in μ_{eff} due to a deviation from the Curie Law:

$$\chi_a = \mu_{\text{so}}^2 / 8(T + \theta)$$

Using your value of χ_a and the formula above, determine a value for θ .

Notice that whereas CaMnO_3 is antiferromagnetic, $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$ and $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ are ferromagnetic. What do your measurements on $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_3$, and $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ tell you about the change in Curie temperature (associated with the onset of ferromagnetic behaviour) with Mn oxidation state in this system?

Superconductors are materials which lose all electrical resistivity below a certain temperature, the critical temperature, T_c ; above T_c , they are generally metallic in nature. Until 1986, when "high temperature superconductors" were discovered, the highest T_c was 23 K for Nb_3Ge . $\text{YBa}_2\text{Cu}_3\text{O}_7$ was the first material discovered with T_c above the temperature of liquid nitrogen, 77 K. When pure, and having its maximum possible oxygen content, this material becomes superconducting at 93 K. An important property of superconductors is that below T_c ,

magnetic fields are expelled from within the material - it becomes a perfect diamagnet. This is achieved by setting up currents on the surface of the bulk material to oppose the applied magnetic field. In this way, it is possible to float a superconductor above strong magnets, using the induced opposing field for levitation.

The structure of $\text{YBa}_2\text{Cu}_3\text{O}_7$ (Fig. 3 and model if available) comprises three perovskite-like unit cells in a row; the Y and Ba ions occupy the large cation positions but in this material, not all the oxygen sites are occupied, which reduces the coordination numbers for all the cations. Layers of 5-coordinate (square pyramidal) and chains of 4-coordinate (coplanar) Cu ions are formed. What is the ratio of 5-coordinate Cu to 4-coordinate Cu in the structure (remember that certain sites in the unit cell are shared with other unit cells)? What is the ratio of Cu^{2+} to Cu^{3+} ions in $\text{YBa}_2\text{Cu}_3\text{O}_7$? A square pyramidal crystal field influences the d -orbital energies of a transition-metal ion in the same way as an elongated octahedral (tetragonal) field, which is common for Jahn-Teller distorted ions. On the basis of Crystal Field Theory, discuss the preferred distribution of the Cu^{2+} and Cu^{3+} ions between the available sites in $\text{YBa}_2\text{Cu}_3\text{O}_7$.

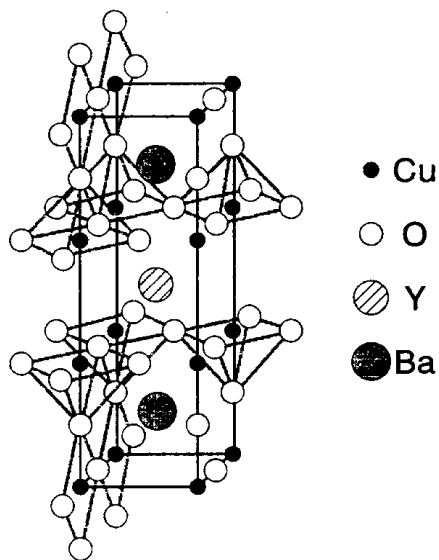
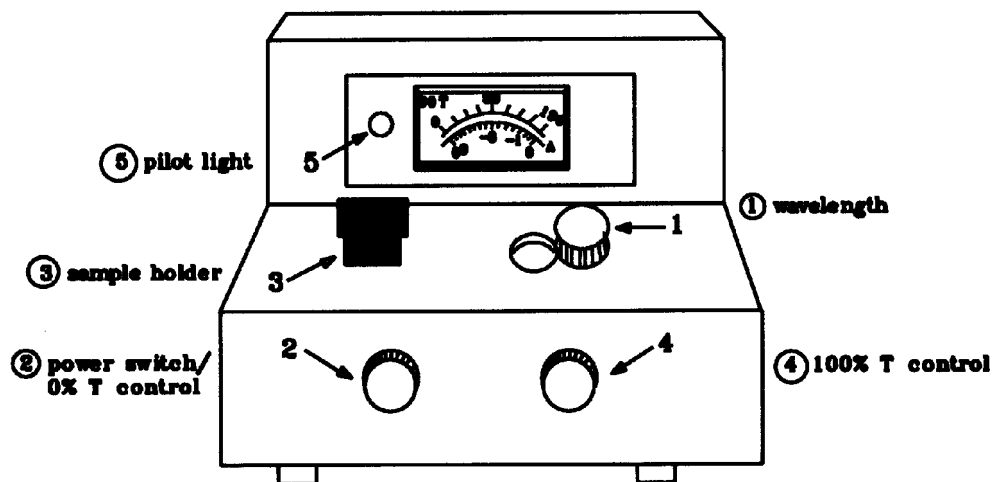


Figure 3. The unit cell of $\text{YBa}_2\text{Cu}_3\text{O}_7$ highlighting the Cu stereochemistry.

NOTES ON THE USE OF THE "SPEC 20"

- A. Turn power switch on and allow 5 minutes for warm-up.
- B. Set the instrument at the desired wavelength.
- C. Adjust the instrument needle to read 0% on the Transmittance scale by turning the lower left-hand knob. Be sure cover of cuvette holder is closed.
Rinse a colorimeter tube (cuvette) with "Blank" solution and then fill about two-thirds its capacity. Wipe and polish the lower half of the cuvette with a tissue. Keep your fingers off the lower part of the tube. Raise the cover and insert the cuvette into the cuvette holder as far down as it will go. The vertically-etched line at the top of the cuvette should line up with the indicator line at the top of the plastic cuvette holder. Close the cover so that outside light does not affect the phototube.
- D. Adjust the needle to read 100% Transmittance by turning the lower right-hand knob.
- E. Remove cuvette containing "Blank" and repeat step 2, readjusting lower left-hand knob if necessary.
- F. Reinsert "Blank" cuvette and repeat step 4, readjusting right-hand knob if necessary.
- G. Rinse, and then fill a second cuvette with the first standard solution. Record the Absorbance reading, estimating to 3 decimal places. Continue with other samples.
Check the settings of the instrument frequently to assure yourself that the needle has not drifted from your original settings made in steps 2 and 4.



NOTES ON THE USE OF THE HI 8819 CONDUCTIVITY METER

General Description

The HI 8819 is a digital benchtop conductivity meter designed for simplicity of use in measuring electrical conductivity in liquids. Four ranges of conductivity measurements are provided with manual temperature compensation. Front panel features a large LCD display with splash proof touch keys. The instrument is operated from AC mains supply.

Each instrument is supplied with a plastic conductivity probe with 1 meter cable length and a dust cover.

The Front Panel

See Figure 1.

Membrane Keys:

- ON/OFF** : Switches instrument on and off.
- COND/TEMP** : Selects the display of conductivity readings or temperature settings for compensation.
- 199.9 μ S** : Selects the range 0.0 to 199.9 μ S.
- 1999 μ S** : Selects the range 0.0 to 1999 μ S.
- 19.99 μ S** : Selects the range 0.0 to 19.99 μ S.
- 99.99 μ S** : Selects the range 0.0 to 99.99 μ S.

Knob:

- TEMPERATURE** : For setting to the temperature of the test solution for temperature compensation.

4. The Rear Panel

See Figure 2

Electrode Connection:

Connect the conductivity electrode to the DIN socket located at the rear of the instrument.

Voltage selector switch:

Set the voltage selector switch to 110V or 220V BEFORE applying power to the equipment.

5. Calibration

Initial preparation:

- If you are measuring in the mS range, calibrate the meter using HI 7030 conductivity solution (12.88 mS at 25°C) or HI 7034 conductivity solution (80 mS at 25 °C) depending on which of the

two mS ranges you intend to use. For the μS ranges, use HI 7031 conductivity solution (1,413 μS at 25°C) when calibrating in the range from 0 to 1999 μS or HI 7033 conductivity solution (90 μS at 25°C) when calibrating in the range from 0 to 199.9 μS .

- Use a thermometer with an accuracy of 1 °C for measuring the temperature of the solution.
- Rinse the electrode thoroughly in distilled water if you have been using the electrode. This is to minimize contamination of the calibration solution.

Procedure:

- Pour a small quantity of the conductivity solution in a beaker.
- Immerse the conductivity probe and thermometer in the solution and wait for therm equilibrium.
- Note the temperature of the conductivity solution.
- Switch the instrument on and press "COND/TEMP" key to display temperature settings.
- Adjust the "TEMPERATURE" knob to display 25 °C if you are using 25 °C as the reference temperature.
- Press the "COND/TEMP" key to display conductivity readings and select the appropriate conductivity range.
- Using a small screwdriver adjust the trimmer on the rear panel until the display shows the conductivity reading at the temperature of the solution noted earlier (see Table of conductivity vs temperature on the solution label).
- The calibration is now complete and the instrument is ready for use.

To check the calibration:

- Press the "COND/TEMP" key to display the temperature.
- Adjust the "TEMPERATURE" knob to display the temperature of the calibration solution, i.e. the temperature noted with the thermometer.
- Press the "COND/TEMP" key to display conductivity.
- The display should read the conductivity of the calibrating solution at the reference temperature.

****NOTE**** The instrument should be recalibrated regularly or whenever the probe has been changed.

6. Conductivity Measurements

Make sure that the instrument has been calibrated before taking conductivity measurements. Connect the probe to the meter and switch the instrument on.

- Take the temperature of the solution with a thermometer.
- Press the "COND/TEMP" key to select temperature mode.
- Adjust the "TEMPERATURE" knob until LCD displays the temperature of the solution.
- Press the "COND/TEMP" key to select conductivity measurements.
- Immerse the probe in the solution.
- If the display shows only a "1", it means an over-range condition in which case the next higher range should be selected.
The conductivity reading displayed has been manually compensated for temperature variations.
- After measurement is completed, the instrument should be switched off and the probe should be cleaned. (see section 7)

7. Probe Maintenance

Rinse the electrode with tap water after every series of measurements.

Some Useful Constants

Constant and symbol	Value
Gas constant, R	1.98722 cal K ⁻¹ mol ⁻¹ 82.0578 mL atm K ⁻¹ mol ⁻¹ 62,363.9 mL torr K ⁻¹ mol ⁻¹ 8.31451 J K ⁻¹ mol ⁻¹
Ideal gas volume, std. cond.	22,414.1 mL mol ⁻¹
Avagadro's number, N	6.022137 x 10 ²³ mol ⁻¹
Faraday constant, F	96,485.3 C mol ⁻¹ 23,060.5 cal volt ⁻¹ mol ⁻¹
Boltzmann's constant, k	1.38066 x 10 ⁻²³ J K ⁻¹
Electron charge, e	1.602177 x 10 ⁻¹⁹ C 4.80321 x 10 ⁻¹⁰ abs. esu
Electron mass, m	9.109390 x 10 ⁻²⁸ g
Proton mass, M_p	1.672623 x 10 ⁻²⁴ g
Planck's constant, h	6.626076 x 10 ⁻³⁴ Js
Newtonian constant of gravity, G	6.6726 x 10 ⁻¹¹ m ³ kg ⁻¹ s ⁻²
Bohr radius, a_0	0.529177 Å
Ice point (0°C)	273.150 K
Speed of light, c	2.99792458 x 10 ¹⁰ cm s ⁻¹

Viewing: Atomic weight

1	Viewing: Atomic weight																18	
1	1 H 1.0079															2 He 4.0026		
2	3 Li 6.941	4 Be 9.0122											5 B 10.811	6 C 12.011	7 N 14.007	8 O 15.999	9 F 18.998	10 Ne 20.18
3	11 Na 22.99	12 Mg 24.305											13 Al 26.982	14 Si 28.086	15 P 30.974	16 S 32.066	17 Cl 35.453	18 Ar 39.948
4	19 K 39.098	20 Ca 40.078	21 Sc 44.956	22 Ti 47.88	23 V 50.942	24 Cr 51.996	25 Mn 54.938	26 Fe 55.847	27 Co 58.933	28 Ni 58.693	29 Cu 63.546	30 Zn 65.39	31 Ga 69.723	32 Ge 72.61	33 As 74.922	34 Se 78.96	35 Br 79.904	36 Kr 83.8
5	37 Rb 85.468	38 Sr 87.62	39 Y 88.906	40 Zr 91.224	41 Nb 92.906	42 Mo 95.94	43 Tc (97.91)	44 Ru 101.07	45 Rh 102.91	46 Pd 106.42	47 Ag 107.87	48 Cd 112.41	49 In 114.82	50 Sn 118.71	51 Sb 121.76	52 Te 127.6	53 I 126.9	54 Xe 131.29
6	55 Cs 132.91	56 Ba 137.33	57 La 138.91	72 Hf 178.49	73 Ta 180.95	74 W 183.84	75 Re 186.21	76 Os 190.23	77 Ir 192.22	78 Pt 195.08	79 Au 196.97	80 Hg 200.59	81 Tl 204.38	82 Pb 207.2	83 Bi 208.98	84 Po (209)	85 At (210)	86 Rn (222)
7	87 Fr (223)	88 Ra (226)	89 Ac (227)	104 Rf (261.1)	105 Ha (262.1)	106 Sg (263.1)	107 Ns (262.1)	108 Hs (265.1)	109 Mt (266.1)	110 Unn (268)	111 Unu (269)							

Lanthanide Series	58 Ce 140.12	59 Pr 140.91	60 Nd 144.24	61 Pm (144.9)	62 Sm 150.36	63 Eu 151.97	64 Gd 157.25	65 Tb 158.93	66 Dy 162.5	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.04	71 Lu 174.97
	Actinide Series	90 Th 232.04	91 Pa 231.04	92 U 238.03	93 Np (237)	94 Pu (244.1)	95 Am (243.1)	96 Cm (247.1)	97 Bk (247.1)	98 Cf (251.1)	99 Es (252.1)	100 Fm (257.1)	101 Md (258.1)	102 No (259.1)