## **PROCEEDINGS**

of the

# NORTH DAKOTA ACADEMY OF SCIENCE

Founded December, 1908

VOLUME XI 1957

### PUBLICATION COMMITTEE

Rae H. Harris (Chairman)
G. A. Abbott
Ernest D. Coon
Ralph E. Dunbar
Ben G. Gustafson

Published jointly by the University of North Dakota and the North Dakota Agricultural College

July, 1957 GRAND FORKS, NORTH DAKOTA

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### **OFFICERS**

President - - - - - - Warren C. Whitman, NDAC
President -Elect - - - - Arthur W. Koth, University
Secretary-Treasurer - - Ben G. Gustafson, University
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W. E. Cornatzer, Ex-officio, University

Walter L. Knudson, School of Forestry H. E. Murphy, Dickinson State Teachers College

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### NORTH DAKOTA ACADEMY OF SCIENCE

### FRIDAY, MAY 3, 1957

President W. E. Cornatzer called the meeting to order at 9 a.m. with 37 members present in the Medical School Auditorium on the University Campus. He greeted those present and made some routine announcements. Then the nine morning papers, (numbers 1 to 8 which were student papers), were read and discussed.

There were some fine scientific exhibits on hand and a coffee break was enjoyed at 10:00 a.m.

The Publications Committee requested permission to ask the assistance of Dean Emeritus Harlow L. Walster in judging the very fine student papers that had been presented. This request was granted.

The first business session was called to order at 11:40 a.m. by President W. E. Cornatzer.

The nominating committee, composed of Dr. E. D. Coon, Chairman, Dr. Ralph Dunbar, Dean R. B. Witmer, Professor Glenn S. Smith and Dr. Harry B. Hart, reported. The following slate of officers were elected for the year 1957-1958:

Chairman—Warren C. Whitman Chairman-Elect—Arthur W. Koth Secy.-Treas.—Ben G. Gustafson Historian—George A. Abbott

Executive Committee:

W. E. Cornatzer, ex-officioWalter L. KnudsonH. E. Murphy

Secretary Ben G. Gustafson reported on the proposed change in the Constitution of The North Dakota Foundation Engineering and Science Scholarships. Lines 1-3 of Section 4 to be changed to read: "The Board of Directors shall be composed of the following: The President, Vice-President or President-Elect and the Secretary and one other selected member of each of the participating groups. The added portion being the term—"and one other selected member." The members were told they would be asked to vote on this proposed change Saturday.

The morning session then adjourned (12:10 noon) to the University Student Union for lunch, the visitors were guests of the University.

The afternoon session was called to order at 1:30 p.m. in the Medical Auditorium where they listened to the invited paper on

"The Physiology of Cold" by Dr. H. E. Ederstrom of the University Medical School Faculty. This paper is reported in the Proceedings.

At 2 p.m. the Academy divided into two sections and papers numbers 11-20 inclusive were read and discussed in the Medical Auditorium with Dr. W. E. Cornatzer presiding. Papers numbers 21-28 inclusive were read and discussed in Room 301 of the Medical Science Building with Dr. Warren C. Whitman presiding.

The Academy Dinner was held in the beautiful Student Union Ballroom at 6:15 in the evening. It was a joint meeting with the Red River Valley Section of The American Chemical Society and the University Chapter of The Society for the Sigma Xi. One hundred twenty four members and guests were present. The meeting was presided over by Dr. Warren C. Whitman, in the absence of Dr. W. E. Cornatzer who had been called to Washington, D. C. to act on a special research panel. Greetings were cordially extended by President George W. Starcher who dwelt on the theme that the University would be celebrating it's 75th Diamond Jubilee in the same year the Academy was celebrating it's 50th Golden Jubilee Anniversary. Professor Edward C. Lawson presented 5 of the 7 winners in the Engineering and Science Scholarship competitions, who were present as guests of the University Alumni Association. Professor Lawson partciularly thanked Dr. E. J. O'Reilly of the University Chemistry Department for sending out and then grading the papers of the 300 High School Seniors who competed, he especially noted that the first 25 in rank were in the upper 8% on a National rating of the examination. Dr. Rae Harris then presented the A. Rodger Denison awards for the Student Research Papers, after describing the difficult job of judging so many fine papers-First place going to Mr. Joseph Siefker, second place to Mr. Alfred E. Decoteau, third place to Mr. Philip H. Harju and Honorable Mention to Mr. Everett E. Wilson. Dr. Harris informed the audience that Dr. A. Rodger Denison had provided for the printing of all the student papers.

Dr. Francis A. Jacobs then introduced the guest speaker, Dr. David E. Green, Chairman of The Institute for Enzyme Research and Professor of Enzyme Chemistry at Wisconsin University. Dr. Green presented a well illustrated and very scholarly report on the "The Structure and Enzymatic Function of the Mitochondrion." The session adjourned at 9 p.m.

### SATURDAY, MAY 4, 1957

The meeting was called to order in the Medical Auditorium at 8:30 a.m. by Dr. Warren C. Whitman. The seven papers, numbers 29-35 inclusive, were read and discussed.

The Annual business meeting was called to order at 10:40 a.m. The minutes of the 1956 Annual Meeting were approved as printed in Vol. X (1956) of "The Proceedings." The Secretary's report was presented and accepted. The Treasurer's report was presented and accepted.

Dr. Herbert J. Fromm reported on the Annual Meeting of the A.A.A.S. held in New York City in December of 1956. The report was accepted. Dr. Fromm used only \$3.00 of the \$25.00 maximum that was set by the 1956 session.

Dr. William L. Downing presented the report of the resolutions committee. This report was accepted and ordered printed in the Proceedings.

### REPORT OF THE COMMITTEE ON RESOLUTIONS

### MAY 4, 1957

- 1. We express our appreciation to President Starcher, to the Administration and the Faculty of the University of North Dakota, for extending its facilities to the Academy and for the luncheons provided.
- 2. We thank the publications of the State for the excellent publicity and coverage that they have given to our meetings.
- 3. We are grateful to the Red River Valley Section of the American Chemical Society, and to the University of North Dakota Chapter of the Society of the Sigma Xi for cooperating in the sponsorship of the informal dinner; and to the Medical School of the University of North Dakota for sponsoring the evening's speaker, Dr. David E. Green.
- 4. We extend an expression of special gratitude to the Administrations of the University of North Dakota, the North Dakota Agricultural College and Jamestown College, for continuing their policy of financing the publication of the proceedings of the Academy and for the large increase they have made in this contribution during the past year.
- 5. We appreciate the fact that editing and publishing the Proceedings is a difficult and time-consuming project; and we wish to thank the Publications Committee for carrying it out so capably.
- 6. We note with regret the passing of Henry J. Bolley, a charter member and one of the original organizers of the Academy.
- 7. To the Officers, the Executive Committee and the other committees of the Academy, we express our thanks for providing the excellent program of papers, the comfortable arrangements, and for the successful conduct of this highly interesting and profitable meeting.
- 8. We wish to express our approval of the system of concurrent sessions on Friday afternoon, and we resolve that this policy be continued in future meetings.
- 9. We are very grateful to those who have so generously contributed to the scholarship fund of the North Dakota Engineering and Science Foundation.
- 10. We urge all members to send names of prospective members to the Membership Committee so that all qualified scientists in North

Dakota may have the chance to make the fullest possible use of the Academy.

11. We express our sincere appreciation to A. Rodger Denison, Vice-President of the American Petroleum Corporation, for continuing his gift for student research prizes, and for paying for the publication of the student papers.

Respectively submitted, George C. Wheeler, C. W. Fleetwood, William L. Downing, Chairman

# REPORT OF COMMITTEE ON SCIENCE LIBRARY FOR HIGH SCHOOLS

In 1955 The American Association for the Advancement of Science and the National Science Foundation assembled a collection of 150 books which were circulated during the school year of 1955-56 among 66 selected high schools in 12 states. In 1956 the list was revised and 200 books were sent to 104 schools in 20 states, including Hettinger, Valley City, Wahpeton and Williston, North Dakota (Science, Dec. 16, 1955 and Nov. 23, 1956).

The project was an experiment to see how much interest could be developed among high school students. Since the books were available to only a few schools for a short time, it seemed that local groups might well assist by providing a few books which would be permanently available to any school. A motion was adopted at the 1956 meeting of the Academy to secure a set of books to the extent of \$50, to be deposited with and circulated by the State Library Commission.

A list was selected by the committee appointed and the books were sent to the State Library Commission about May 1, 1957. Each book was marked as follows:

Presented by
the
North Dakota
Academy of Science
Take care of books
They are your friends

O. A. Stevens, chairman

After considerable and a somewhat heated debate it was moved by Ben G. Gustafson that the Executive Committee be instructed to relieve the "Publications Committee" of the job of judging the Student Research Papers, and the Executive Committee be instructed to prepare a method of judging that would be equitable and satisfactory. The motion was seconded by Rae H. Harris and was carried.

### REPORT OF THE COMMITTEE ON NECROLOGY

Dr. O. A. Stevens gave the report of the Neurology Committee which was accepted and ordered printed in The Proceedings.

### Henry Luke Bolley

H. L. Bolley, one of the most widely known North Dakota scientists, died on November 9, 1956. One ofthe first members of the Agricultural College staff in 1891, he was always a leader. At first he taught both botany and zoology and gave attention to milk sanitation, typhoid fever and tuberculosis. Treatment for potato scab was one of his early accomplishments, and he began potato certification, which has since become an important asset to the state.

When the present writer first traveled in North Dakota he was surprised that farmers recognized Bolley's work in controlling wheat smut but thought little of his work on wilt resistant flax which was to be the chief interest of his later years. Many varieties, more or less resistant, were sent out and replaced by better ones when they became available. A colleague has called him "The Grand Old Man of Flax."

He was largely responsible for securing legislation to eradicate the common barberry and thus reduce wheat losses from rust. He secured passage of a seed control law to check the sale and planting of noxious seeds, and administered it for 20 years without bringing a case to court. Education and persuasion were his arms. Chemical control of weeds was one of his early studies.

He had a wide acquaintance with farmers and others throughout the state. He felt that he had not finished a project until it reached the farmer. He wrote some 20 bulletins, 48 circulars and "press bulletins," 66 technical articles besides hundreds of articles for farm papers.

### O. A. STEVENS, Chairman

Dr. Fancis A. Jacobs moved the proposed change in the Constitution for "The North Dakota Foundation for Engineering and Science Scholarships" be approved. The motion was seconded by Dean Harlow L. Walster and was carried. Section 4 was changed to read "The Board of Directors shall be composed of the following: The President, Vice-President or President-Elect, the Secretary and one other selected member of each of the participating groups." The manner of selection was left to the Executive Committee for the Academy.

Dr. H. E. Ederstrom then reported for the Membership Committee. Upon recommendation of the committee sixty-nine were elected to active membership subject to payment of their annual dues before June 1, 1957. Their names are given in the list of active members in The Proceedings.

The Academy adjourned it's 49th session at 12:15 Noon and went to lunch at the Student Union, visitors were guests of the University.

Respectively submitted, Ben G. Gustafson Secretary

# A SPECTROPHOTOMETRIC STUDY OF THE DISSOCIATION OF DINITROGEN TETROXIDE

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### INTRODUCTION

The dissociation of dinitrogen tetroxide,  $N_2O_4 = 2NO_2 = 2NO + O_2$ , has been studied by numerous investigators by vapor density or pressure methods.<sup>12</sup> These workers assumed that the first dissociation,  $N_2O_4 = 2NO_2$ , was complete before the second dissociation,  $2NO_2 = 2NO + O_2$ , began. That is, they made the assumption that when the vapor density of the gas or the pressure exerted by it corresponded to that expected for nitrogen dixoide alone, the dissociation represented by the equation,  $N_2O_1 = 2NO_2$ , was shifted completely to the right.

Since  $NO_2$  is a highly colored brown gas and  $N_2O_1$  and NO are colorless gases, a colorimetric method for the determination of the degree of dissociation of dinitrogen tetroxide should be practicable. The brown gas should absorb light in the visible, while the other gases should not. That is, the amount of absorption of light should be directly proportional to the concentration of  $NO_2$ .

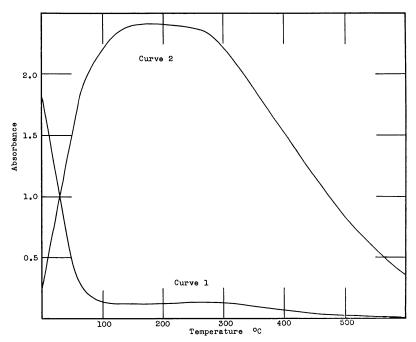
T. C. Hall, Jr., and F. E. Blacet have resolved the absorption spectra of nitrogen dioxide and dinitrogen tetroxide.<sup>3</sup> They reported that in the ultraviolet at 2500 A the absorption is due primarily to dinitrogen tetroxide and that between 2500 A and 4000 A the amount of absorption due to nitrogen dioxide increases, while the amount due to dinitrogen tetroxide decreases. In the visible between 4000 A and 5000 A they reported the absorption to be completely due to nitrogen dioxide.

The present investigation has included determinations in both the visible and the ultraviolet. Curve 1 shows a typical run made in the ultraviolet at 2500 A. The absorption of light decreases very rapidly indicating the disappearance of dinitrogen tetroxide as the temperature rises. The small amount of absorption noted after the curve levels off is probably caused by the dissociation products. Curve 2 shows a typical run made in the visible at 5000 A. The absorption of light increases rapidly at first indicating the formation of colored dissociation products. It then levels off and finally decreases rapidly indicating the further dissociation to colorless products, NO and O<sub>2</sub>.

These findings tend to confirm that dinitrogen tetroxide shows no absorption in the visible although it absorbs strongly in the ultraviolet and that nitrogen dioxide absorbs strongly in the visible and weakly in the ultraviolet. The absorption of light by the gaseous dissociation products of dinitrogen tetroxide has been measured when the temperature of the gases was varied from zero to 660 degrees centigrade.

### **EXPERIMENTAL**

Since such a wide temperature range was used, the spectrophotometer had to be modified somewhat. The basic instrument was a Beckman "Model DU" Quartz Spectrophotometer. A Beckman power supply was used in conjunction with a six-volt storage bat-



tery to assure maximum stability of the intensity of the input light. A tungsten lamp was used as the light source for work in the visible, while a hydrogen lamp was used in the ultraviolet.

The cell compartment was removed and the spectrophotometer was fitted with a specially constructed oven. The insulating properties of the oven had to be good enough so that heat was conducted neither to the body of the instrument nor to the photoelectric cell compartment. This was somewhat of a problem since the lamp house of the spectrophotometer was mounted in such a position as to limit the thickness to which the walls of the oven could be made.

The oven was constructed from  $\frac{1}{4}$ " sheeting of asbestos composition and various cements. The first cement used was a commercially available product. After a time some of the cement cracked

and the oven was rebuilt using a paste made by mixing flint, SiO<sub>2</sub>, with technical grade water glass, a concentrated solution of Na<sub>2</sub>SiO<sub>3</sub>. This cement proved somewhat better; however, when it became necessary to rebuild the oven again, a paste made by mixing black CuO with water glass was used and was found to be the best.

The oven was constructed as a nearly square box, the outer measurements being 5 x 5¾ x 6¾ inches and the inner measurements being 3 x 3 x 4½ inches. The inner dimensions of the oven were large enough so that the gas cell could be moved back and forth, thereby placing the reference cell in the beam of light in one extreme position and the sample cell in the beam of light in the other extreme. The space between the inner and outer walls of the oven was filled with vermiculite as an insulating material. Further insulation was provided by placing blocks of solid MgCO3, 11/8" thick, and water jackets, or thermospacers as they are called by Beckman, on either side of the oven between the oven and the body of the instrument and between the oven and the photoelectric cell compartment. Cold tap water was circulated through the thermospacers and the parts of the instrument near the oven were thereby kept at room temperature or slightly below. This prevented any thermal effects on the photoelectric cells and prevented any expansion of the mirror mountings within the spectrophotometer as the oven became hot.

Quartz windows which allowed the transmission of both visible and ultraviolet light were placed on each side of the oven to prevent circulation of air in the oven by convection currents. The cover for the oven was made by constructing a thin shell of the CuO-water glass cement around a center filled with vermiculite. This construction made the cover light enough to allow easy handling and yet strong enough to prevent breakage. Both the cover and the side of the oven near the opening were notched to provide a guide in which the cell could slide.

The heating element was constructed from B. and S. No. 26 chromel wire and was placed in the ends and the bottom of the cavity of the oven. The first element designed was embedded in cement and placed only in the ends of the oven opening. This design proved to be unsatisfactory since the wire "burned out" frequently. The wires were then made longer and placed in both the ends and the bottom of the cavity and were left exposed to the atmosphere. This design was satisfactory; however, a later improvement was made by embedding the wires in a thin layer of the CuO-water glass cement.

The temperature of the oven was controlled by varying the input voltage with an Acme Electric Corporation "'Voltrol" voltage regulator. The maximum voltage applied was 60 volts which allowed the attainment of 660 degrees centigrade.

The temperature was measured with an iron-constantan thermocouple connected to a Leeds and Northrup galvanometer. Readings were made on a 100 cm. plastic scale graduated in mm. The junctions of the thermocouple were fused with an electric arc. The reference junction was kept at zero degrees in an ice-water bath, while the other junction was placed inside the reference cell in the oven. A cell identical to the sample cell was used as a reference cell. This cell was held in position with a specially made ceramic holder and was filled with air as a reference gas. The air in the reference cell was assumed to be at the same temperature as the gases in the sample cell.

The thermocouple was calibrated using seven thermometers—minus 10- plus 60, 40·100, 90-160, 150-210, 200-260, 250-310, 300-360—each graduated in 0.2° C, and four fixed points—the melting points of tin, lead, antimony, and zinc. The thermocouple was accurate to within 1° C.

The gas cells used were made by the American Instrument Company. One was constructed entirely from pyrex glass, while the other was made from fused quartz. The length of the cell through which the light passed was 50 mm. in both cases.

The cells were wrapped in metals to prevent the admission of stray light from the red-hot oven into the photoelectric cell compartment. A considerable amount of difficulty was experienced with some of the metals. A yellow coloration was left by silver when the cell had been heated to only 400 degrees. The vapor pressure of silver is apparently great enough at this temperature to allow a thin layer of silver to sublime and condense on the glass. Jo Kinumaki and Taro Ito have reported that silver diffuses into glass in the 500-600 degree range. They measured the extent of diffusion by means of a tracer technique using radioactive silver.

Iron was found to be a fairly suitable material for use with glass but not with quartz. A white deposit which could be removed only by mechanical grinding of the surface was left when the quartz cell had been heated to 600 degrees in contact with iron. When the cells were wrapped in platinum and heated to 660 degrees the optical quality of the quartz was not affected. A holder made of fired ceramic clay was used to keep the reference cell, sample cell, and platinum wrappers in position.

At 660 degrees a binder was baked out of the asbestos sheeting of the oven. This left a troublesome deposit on the cell. This trouble was eliminated by coating the inner walls of the oven with a thin layer of the CuO-water glass cement.

"Teflon" plugs coated with a thin layer of Dow Corning high vacuum silicone stopcock grease were used in the stopcocks of the cells. Both the "Teflon" plugs and the silicone grease were found to be inert to the highly reactive grease during the course of a run which was about five hours.

### RESULTS

The primary purpose of this paper is to give a detailed descrip-

tion of the modification of a spectrophotometer by the adaptation of an oven that can be used at temperatures as high as 660 degrees centigrade. This apparatus has been used to study the dissociation of dinitrogen tetroxide with a considerable amount of success. Only the more important findings are now presented; however, a detailed account of the results will be published at a later date.

The runs were made by using a specific wave length and varying the temperature. Various wave lengths in the range of 2500 A to 6630 A were used. Preliminary runs made at 6630 A showed that the maximum absorption occurs at about 320 degrees, while, according to pressure measurements, dinitrogen tetroxide is completely dissociated into nitrogen dioxide at about 140 degrees. "," If the assumptions that nitrogen dioxide is the only absorbing species and that the absorption coefficient for it remains constant are correct, then the maximum concentration of nitrogen dioxide would be expected at 320 degrees.

Runs were then made at wave lengths of 3200, 3500, 3800, 4000, 5000, 5820, and 6150 A and two interesting observations were made. It was found that maximum absorption occurs at 4000 A and also that the temperature at which the maximum absorption occurs varies with the wave length used. This led the observers to believe that two colored gases having different absorption coefficients must be formed when dinitrogen tetroxide dissociates. A postulate was made that the relatively flat portion of the curve (see curve 2) was caused by the simultaneous disappearance of one of the colored species and formation of the other.

The further postulate was made that the second colored gaseous species was either  $NO_3$  or  $N_2O_3$ —these being formed by reactions such as the following:

Equations were developed from both postulates and attempts were made to fit them to the absorption curves. The absorption coefficients for both  $NO_2$  and the other colored gas were assumed to be constant at all temperatures studied.

The equation derived on the assumption that  $N_2O_3$  is the second colored gas seems to fit the curve. The concentrations of the various dissociation products of dinitrogen tetroxide as calculated by this equation will be published later.

### SUMMARY

- 1. A detailed description is given of the modification of a spectrophotometer by the adaptation of an oven that can be used at temperatures as high as  $\tilde{6}60$  degrees centigrade.
- 2. Dinitrogen tetroxide was found to absorb light strongly in the ultraviolet, while nitrogen dioxide was found to absorb strongly in the visible.

- 3. Dinitrogen tetroxide was found to dissociate into some other colored gaseous species which also absorbs light in the visible.
- 4. Serious doubt has been cast on the validity of the assumptions made in the vapor density studies on the oxides of nitrogen.

### ACKNOWLEDGEMENT

The authors are very grateful for the support received from a research fellowship made possible by an Army Ordnance grant.

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# AN INVESTIGATION OF THE CORALS FROM THE CANNONBALL FORMATION (PALEOCENE) OF NORTH DAKOTA

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University of North Dakota

### ABSTRACT

In 1920 T. W. Vaughan described six species of fossil hexacorals from the Cannonball formation near Leith, Grant County, North Dakota. Until a field party of the North Dakota Geological Survey, with F. D. Holland, Jr. in charge and A. M. Cvancara and R. G. Monroe as assistants, discovered a collecting locality near Mandan, Morton County, in June of 1955, no known systematic collecting of the corals had been carried on by any institution or student known to the writer. The collection started by that party has been added to several times with specimens from the Mandan locality and another about 5.5 miles southwest of Leith.

The Cannonball formation crops out in scattered areas on both sides of the Missouri River, in Burleigh, Morton and Grant Counties, and along the Mouse River near Velva, McHenry County, and in a semi-circular strip around the southern end of the Turtle Mountains. Its age has been controversial since it was originally distinguished as a marine member of the "Lance" formation (questionably Eocene) by Lloyd and Hares in 1915. Stanton, in 1920, thought the "Lance" and included Cannonball formation Cretaceous in age after he studied the mollusks of the Cannonball formation. Dorf was the first to use the Cannonball formation as Paleocene in age in 1940; Fox and Ross

added support to this by showing that Forminifera from the Cannonball formation are closely related to Texas Midway (Paleocene) forms. The Paleocene age for the Cannonball formation is now generally accepted.

The types of fossils known to occur in the Cannonball formation include Foraminifera, the corals, mollusks and sharks teeth. Recently Holland and Cvancara (1955) have shown that crabs are also present. Only some of the mollusks and sharks teeth were found with the corals.

The corals are found in an olive gray siltstone unit that weathers to light olive gray chips and flakes. On this flaky surface the corals appear within a day or two after each rainfall. Since they are composed of calcium carbonate they are easily destroyed by weathering. Hence, the best specimens for study, the ones with well preserved pali, costae, septa, basal scars and shapes, should be collected as soon as possible after they reach the surface.

Of the six specimens described by Vaughan in 1920, five are present in this collection. These include Trochocyathus neumani, Paracyathus lloydi, Paracyathus thomi, Paracyathus kayserensis and Bathycyathus leithensis. One new species of Trochocyathus is represented by three specimens. This is a small, distinctive coral with a patellate shape. Another specimen is questionably identified as Caryophyllia sp.

In this study it is significant that a new coral locality has been found, the first in over 30 years, and that one genus and one new species are recognized for the first time in North Dakota.

### THE ACETYLATION OF IMIDES WITH KETENE

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Previous studies in our laboratories have involved the acetylation of alcohols, mercaptans, carboxylic acids, glycols, polyhydroxy compounds, amides, hydrocarbons, and carbohydrates with ketene. All of the above classes of compounds are similar in that they contain activated or readily replaceable hydrogen. Ketene is characterized by its ability to form combinations with such organic substances. Although somewhat toxic and difficult to prepare, it ranks first in reactivity among the common acetylating reagents. The predominating advantage of ketene, as compared to acetic acid, acetyl halides, and acetic anhydride, is the fact that it theoretically produces no objectionable by-products.

Since amides have been found to be readily acetylated with ketene to form mono and diacetyl derivatives, it seemed highly probable, by analogy, that imides could be similarly acetylated to the corresponding acetyl derivative. In reality the monoacetyl derivative of an amide is identical in structure with an imide, insofar as the arrangement of the nitrogen, adjacent hydrogen and two carbonyl groups are concerned. For these reasons it seemed desirable to attempt a similar acetylation study of any and all available imides with ketene

### **EXPERIMENTAL**

The ketene for this study was prepared by the use of a Hurd type "lamp" (1). It was prepared by the pyrolysis of purified acetone. The acetone was vaporized by boiling, and the vapors were passed over an electrically heated wire. The ketene was formed by a free radical breakdown of the acetone. It was mixed with the compound to be acetylated in an absorption apparatus (2). The compound being acetylated was dissolved in a suitable solvent placed in the absorption apparatus.

The imides for this study were all suspended in carbon tetrachloride at room temperature. Succinimide, however, was suspended in hot carbon tetrachloride. A common catalyst found to be effective with all the imides acetylated was sodium acetate. The imide was weighed out in a 0.03 mole quantity. This, together with 0.03 grams of catalyst, was added to an eighty milliliter portion of the solvent in the absorption flask. Ketene was passed through the re-

TABLE 1

Ketene Acetylation Products of Imides					
Imide	Acetylated	M P. $\phi$	M. P.φ	% Nit	rogen
Employed	Appearance	Observed	Mixed	Calculated	Observed
	butterscotch				
Phthalimide	crystals	128- 30°	128- 30°	7.41	7.30
O-Benzoic Sulfimide (saccharin)	white crystals	196-200° (decomp.)	196-200°	6.23	6.25
Pyromellitic Diimide	light tan crystals	138- 40° (decomp.)		9.34	9.31
Succinimide	clear yellow oil			9.93	9.82
Tetrahydro- Phthalimide	light brown crystals	130- 32°		7.77	7.71
4-Nitro- phthalimide $\phi$ Melting Po	bright yellow crystals ints Corrected	105- 07°			

action mixture until an equivalent molar amount had been added. A slight excess was added to insure complete reaction. After the addition was complete the mixture was separated from any residue which

failed to dissolve. This residue was found to be the parent compound, and was discarded. The mixture was cooled and the acetylated imide crystallized out in surprisingly pure form.

All acetylated imides were verified using mixed melting points with samples prepared by other means and also with micro-Kjeldahl and micro-Dumas nitrogen determinations.

### RESULTS

Six compounds have been acetylated to date. The results are tabulated in Table 1.

### DISCUSSION

The first difficulty encountered in this study was the selection of a suitable inert solvent. Among those employed were carbon tetrachloride, benzene, ether, ligroin, dioxane, and chloroform. For the imides hot benzene seemed to be the most efficient solvent but also induced excessive polymerization. Dioxane appeared to be a better solvent but could not be readily removed without causing the acetylated imide to decompose. This was most probably due to the formation of peroxides as the dioxane was exposed to air. Also acetylation was extremely slow when this solvent was employed. A suspension of the imide in carbon tetrachloride yielded maximum acetylation with little or no polymerization.

It was found that the imides would not acetylate when reacted with ketene alone. A catalyst was therefore necessary. Concentrated sulfuric acid has been found by other investigators to be a very satisfactory catalyst in other studies. When concentrated sulfuric acid was used with the imides no acetylation took place. By melting point techniques it was found that there was no change in the melting point from that of the parent compound either before or after the acetylation. Fused sodium acetate produced positive results. The compounds which were unaffected by the ketene in previous attempts acetylated readily with no by-products when sodium acetate was used. This catalyst was found to be satisfactory with every imide treated.

A unique case arose with the acetylation of succinimide. When acetylated, using hot benzene as solvent, a tarry polymer formed which could not be readily identified. Upon further search of the literature it was found that carbon tetrachloride caused the least polymerization of any solvent commonly used in ketene acetylations (4). This solvent was used with gratifying results. The succinimide did not dissolve in the carbon tetrachloride but remained suspended in the hot solvent. As the reaction progressed an oil formed. When an equivalent amount of ketene had been added it was found that all of the succinimide—crystals had reacted to form a brownish oil, less dense than the carbon tetrachloride. The oil darkened upon standing in the solvent. This oil was removed from the solvent and purified by vacuum distillation at 2 millimeters and 122 degrees.

The structure of the acetylated succinimide was proved, in addition to Nitrogen determination, by a reaction forming acetanilide. An equivalent amount of N-acetyl succinimide was added to aniline. An exothermic reaction resulted and a dark oil was formed. When this oil was poured into water acetanilide formed. This was recrystallized. By calculating the theoretical and actual yield of this reaction it was definitely established that the N-acetyl compound was formed (3).

This work could be valuable from the standpoint of the organic analytical student because derivatives of imides are as yet little known. The literature is very sketchy concerning imide derivatives and they are difficult to prepare. The acetyl derivatives of imides have previously been prepared by reaction with acetic anhydride followed by long hours of refluxing to affect a reaction. The yields are low. Attempts to acetylate imides using acetic acid or acetyl halides have been unsuccessful. Ketene will react readily with the imides producing satisfactory yields in a matter of minutes. The gas can be produced readily on a laboratory scale and is becoming of increasing importance commercially.

On the commercial scale N-substituted imides have been used successfully as insecticides and textile water repellents. It appears that these N-acetyl imides could be employed in these fields, as soon as their preparation has been perfected.

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### MYRISTIC ACID AND RELATED COMPOUNDS

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### ABSTRACT

Trimyristin was obtained as a student preparation by the Soxhlet-ether extraction of nutmegs. The trimyristin was converted to myristamide by heating with NH<sub>4</sub>OH (conc.) in a sealed tube at 200° C. The amide was converted by way of the first of a two-step Hofmann hypobromite degradation reaction to methyl tridecylcarbamate. The direct single-step conversion to the amine is not feasible. Myristamide was also prepared from myristic acid by way of myristoyl chloride. Methyl myristate was obtained by Fischer esterifica-

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tion of myristic acid (as well as by ester-interchange of methanol and trimyristin).

### INTRODUCTION

As a result of a student preparation of trimyristin from nutmegs (1, 2, 3), I became interested in the chemistry of the moderately long chain aliphatic compounds and decided to run the standard types of aliphatic reactions, including chain-building and degradation, on these longer chains rather than on the 2 to 5 carbon compounds as is usually done. After consultation with my instructor, he approved of my idea, and together we arranged a program of reactions to be carried out. A study of the literature indicated that some of the reactions proceeded somewhat differently for the long-chain than for the short-chain compounds. While we have not prepared any new compounds, we have prepared a few by new or modified procedures for the given compound.

### **EQUATION FOR REACTIONS**

I from Myristica officinalis, moschata, fragrans, surinamensis (5), etc.

(n-C<sub>13</sub>H<sub>27</sub>COO)<sub>3</sub>C<sub>3</sub>H<sub>5</sub> + 3NH<sub>3</sub> (aq.) + heat—→
3n-C<sub>13</sub>H<sub>27</sub>CONH<sub>2</sub> + CH<sub>2</sub>OH-CHOH-CH<sub>2</sub>OH

II Myristamide Glycerol

II Myristamide Glycerol

3n-C<sub>13</sub>H<sub>27</sub>COOH + PCl<sub>3</sub>—→3n-C<sub>13</sub>H<sub>27</sub>COCl + P(OH)<sub>3</sub>

III Myristic Acid IV Myristoyl Chloride
n-C<sub>13</sub>H<sub>27</sub>COCl + 2NH<sub>3</sub> (aq.)—→n-C<sub>13</sub>H<sub>27</sub>CONH<sub>2</sub> + NH<sub>1</sub>Cl

II Myristamide

 $(C_{13}H_{27}COO)_3C_3H_5$  Trimyristin from nutmeg butter (1, 2, 3, 4, 5);

# DISCUSSION OF THE HOFMANN HYPROBROMITE DEGRADATION OF AN AMIDE TO AN AMINE (6)

For the lower n-aliphatic amides up to 8 carbon atoms, the Hofmann hypobromite amide degradation reaction takes place in accordance with the equation:

$$\begin{array}{lll} C_nH_{2n+1}CONH_2 \ + \ Br_2 \ + \ 4NaOH & \longrightarrow \\ C_nH_{2n+1}NH_2 \ + \ 2NaBr \ + \ Na_2CO_3 \ + \ 2H_2O \end{array}$$

The mechanism of this reaction involves the intermediate formation of an N-bromo-amide and of an isocyanate.

In the dry state, the salt ion undergoes a decomposition and rearrangement,

$$(RCONBr)^-\longrightarrow (RC(=O)N=) + Br^-$$
  
 $(RC(=O)N=) \longrightarrow O=C=N-R,$ 

the product being an isocyanate. In the presence of water and an excess of alkali, the isocyanate is hydrolyzed to an amine.

$$R-N=C=O + OH^- \longrightarrow (RNHCOO)^-$$
  
 $(RNHCOO)^- + OH^- \longrightarrow RNH_2 + CO_3=$ 

In alcoholic solution, the isocyanates yield urethans.

$$R-N=C=O + C_2H_5OH \longrightarrow RNHCOOC_2H_5$$

The urethans can be converted to the amines by heating with slaked lime.

$$RNHCOOC_2H_5 + Ca(OH)_2 \longrightarrow RNH_2 + CaCO_3 + C_2H_5OH$$

When one-half of the usual quantities of bromine and alkali are employed, the isocyanate and the halo-amide ion react to give an alkyl acyl urea. This on hydrolysis gives back one-half mole of aliphatic acid or amide, one-half mole of amine degradation product, and one-half mole of CO<sub>2</sub>.

$$H_2O + RN = C = O + (RCONBr)^- \longrightarrow RNHCONHCOR \xrightarrow{H_2O} RNH_2 + RCONH_2(OH) + CO_2 + (NH_3).$$

### **EXPERIMENTAL**

### Trimyristin (I)

Trimyristin (1) was extracted from nutmegs with ether, using a Soxhlet extraction apparatus, and then recrystallized from methanol. The snow-white needles melted at 55° C. (Richter reports 56.5° C.)

### Myristamide (Tetradecanamide) (II)

Trimyristin was converted into myristamide by the method of Lutz (9). 1.00 g. of trimyristin was placed in a micro-Carius tube along with 2 ml. (0.115 mole) NH.OH (7 fold excess). The tube was sealed and placed in an electric oven at 200° C. for 20 hours. The tube was opened and the contents were disolved in ether and the products, myristamide and gycerol, were separated. The melting point of the myristamide, which was recrystallized from methanol twice, was 97-102° C. (Lutz, 102° C.)

### Myristoyl chloride solution (IV)

This was prepared by a procedure given for capryl chloride (10). Redistilled PC1<sub>3</sub>, 20 g. (12.9 ml.), was added to a solution of 40 g. myristic acid (III) in 150 ml. reagent-grade n-hexane (b. p. 68° C.). The mixture was allowed to stand for one-half hour and then was refluxed for one hour.

### Myristamide (II)

The myristoyl chloride solution above was poured directly (with

continual agitation) into 200 ml, of conc.  $NH_1OH$  cooled in ice. The precipitate was filtered off from the two-phase solution, washed with 10 ml. cold hexane, and then with 25 ml.  $H_2O$ , and recrystallized from 150 ml. of acetone. The white needles melted at  $102^{\circ}$  C. The yield was 38.2 g., or 95.6% as based on myristic acid.

### Methyl myristate (VI)

Methyl myristate (methyl tetradecanoate) was prepared by a modification of a procedure for methyl benzoate (11). A mixture of 25.0 g. myristic acid (III), 100 ml. methanol, and 5 ml.  $\rm H_2SO_4$  was refluxed for two and one-half hours. After distilling off the excess methanol, the ester was taken up in ether, washed with 10% Na bicarbonate, and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. The clear solution was decanted off and the ester distilled over at 293-7° C. (corr.) at 747 mm. (Beilstein (12), 295° C. at 751 mm.) Methyl myristate occurs in iris roots and in the aroma of violets (13).

### Methyl tridecylcarbamate (6) (V)

The procedure used was a slight modification of the procedure given for the preparation of methyl pentadecylcarbamate from palmitamide by the first step of a two-step Hofmann hypobromite degradation (6). A sample of 0.91 g. (0.0042 mole) of myristamide (II) was placed in solution with 20 ml. of methanol. To this was added a solution of 0.23 g. (0.01 gram atom) of metallic Na in 7 ml. of methanol. 0.64g. (0.004 mole) of Br<sub>2</sub> was then added with thorough mixing. The solution was heated for 10 min. on a steam bath, after which it was made just acid with acetic acid. The methanol was evaporated off and the product was washed with water. The product (methyl tridecyl-carbamate) was then dissolved in ligroin and the product was recrystallized twice from 75% methanol-25% water solution. The melting point was 47-50° C. The percent yield was about 75. (Lutz (9), m.p. 53-4° C.).

### ACKNOWLEDGEMENT

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### EXOGENOUS PHASES IN THE LIFE CYCLE OF NEMATODIRUS ABNORMALIS, A NEMATODE PARASITIC IN SHEEP

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### INTRODUCTION

As early as 1915 at least one life cycle of a nematode in the genus Nematodirus was known. Boulenger studied the free living stages and some parasitic stages of Nematodirus filicollis which were obtained from naturally infected sheep. It was not until 1951, however, that Crofton and Thomas discussed another species, Nematodirus battus, which was considered quite detrimental to the sheep of Great Britain. In further studies, Herlich and Porter (1953) investigated the life cycle of Nematodirus helvetianus, a nematode parasitic in cattle. Turner (1953) studied the survival of eggs and larvae of Nematodirus spathiger after exposing them to various laboratory and pasture conditions. Kates and Turner (1955) discussed the complete life cycle of this nematode.

The objectives of this paper are to present a brief description of the exogenous phases of the life cycle of **Nematodirus abnormalis** and to compare these observations with those of other workers on species of the same genus.

### MATERIALS AND METHODS

The main supply of eggs were passed in the feces of a lamb which was naturally infected with this and other nematode species. It was necessary to remove the eggs from the feces. Seghetti (1955) utilized various sizes of wire mesh sieves to separate **Nematodirus** 

spathiger ova from other species and fecal debris. A similar method was employed in which approximately three hundred grams of finely mashed feces were mixed with 1000 grams of tap water. This mixture was then passed through a 60 mesh U.S. Standard sieve with the aid of running tap water. The residue in the sieve was discarded; the filtrate was allowed to settle. After decanting, the settled filtrate was then placed in a 100 mesh sieve. Tap water was run through the material and the filtrate was again allowed to settle. The settled filtrate was placed in a 200 mesh sieve and a forced stream of tap water was utilized to remove any material that would pass through the screen. This operation removed all nematode eggs, other than Nematodirus abnormalis, which remained in the sieve. The 200 mesh sieve was then inverted over a large glass container and the residue in the sieve was washed into the container and allowed to settle. After twenty-four hours, the liquid was siphoned off leaving a sediment containing large numbers of eggs. During the entire separating process the temperatures to which the eggs had been subjected ranged from 10 to 28° centigrade.

The sediment containing the ova was placed 2 millimeters in depth in Petri dishes. To this, 2 millimeters of water were added. This mixture was incubated at 30° (-2) centigrade. Each day some of the incubating eggs were removed with a medicine dropper and placed on a microscope slide for observation.

Additional fecal material was collected from the lamb for the purpose of ascertaining the size of the nematode eggs in fresh manure. Measurements were made on 100 ova. Following this the lamb was slaughtered and all **N. abnormalis** female worms were removed. These were finely ground in a mortar with the aid of a pestle and sterile sand, thus freeing the eggs from the uteri. The ground material was placed in Petri dishes to which 2 millimeters of water had been added and was incubated at  $30^{\circ}(\pm 2)$  centigrade. Observations were made of the development of the eggs at twenty-four hour intervals.

### RESULTS

The eggs from fresh feces were ovoid and the central mass was easily distinguished. The majority of the eggs were in the six to eight cell stage of cleavage. They varied in length from 156 to 236 microns with a mean length of 208 microns. Variation in width was from 84 to 112 microns with a mean width of 103 microns.

After 36 hours of incubation the eggs removed from the female worms had advanced to the blastula stage while no change was noted in those that had been washed from the feces. It was not until the sixth day that these eggs had reached the blastula stage. At this time most of the eggs removed from the female worms were in the late segmentation stages. Others had advanced to the first larval stage. It was not until the fifteenth day of incubation that first stage larvae appeared in cultures of eggs that had been sep-

arated from the feces. In all instances the first stage larvae moved sluggishly within the egg and each contained a disorganized, granular substance.

Toward the end of the eighth day of incubation the larvae cultured from eggs removed from the female worms, became active and commenced writhing. A discarded cuticle was noted, indicating that a second stage larva had developed. The intestinal tract became differentiated by the appearance of a dark cellular mass. On the ninth day the second stage ceased activity and the second molt began. On the thirteenth day of incubation, third stage larvae with definite sheaths began hatching from the eggs. The hatching process continued until the fifteenth day when all eggs had apparently hatched. The infective larvae were very active in the water and swam with an undulating, snake-like motion.

It was not until the seventeenth day that the first stage larvae, cultured in eggs removed from feces, became active and developed into second stage larvae as evidenced by molting. The larvae began their second molt on the eighteenth day of incubation. Hatching of the third stage infective larvae did not occur until the twenty-second day and was evidently complete on the twenty-sixth day. A large number of eggs did not develop beyond the six to eight cell stage.

### DISCUSSION AND CONCLUSIONS

From data presented here it seems that Nematodirus abnormalis ova are slightly larger than N. helvetianus, N. filicollis, and N. spathiger ova. The preparasitic development of all these species appears to be identical under optimum conditions. Differences in the time of development to the infective, or third stage among the species were probably due to differences in temperatures to which the cultures were exposed rather than to differences in embryological development. It is interesting to note that all species of Nematodirus described thus far do not leave the egg until the third or infective stage is reached. This accounts for the high degree of resistance of the species found in this area to our rigorous climate.

Temperature conditions surrounding the eggs previous to larval development exert a pronounced effect on the survival and length of time required for development. Low temperatures profoundly extend this time and also reduce the reproductive potential to a considerable extent. The ability of the nematode to survive these hazards may account for the heavy infections of **N. abnormalis** that occur in sheep of North and South Dakota. This species has not been reported from any other area.

### SUMMARY

The preparasitic development of Nematodirus abnormalis has been studied and has been found to be essentially the same as the development of N. filicollis, N. helvetianus, and N. spathiger. How-

ever, the ova of this species are larger than those of the other species. They are therefore larger than the ova of any species of parasitic nematode. The larvae undergo two molts within the egg and retain the cuticle of the second stage larvae as sheaths when they hatch.

The length of time required from six or eight cell stage to hatching is largely dependent on temperature encountered before larval development. Eggs cultured from fecal material that had been washed with water at temperatures as low as 10° centigrade were slower in development when cultured at an optimum 30° centigrade than were eggs that were removed from female worms and cultured at the same temperature. The initial low temperature also reduced the number of eggs that advanced beyond the six or eight cell stage.

### **ACKNOWLEDGEMENTS**

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### REACTION STUDIES OF ACID HALIDES AND ETHERS

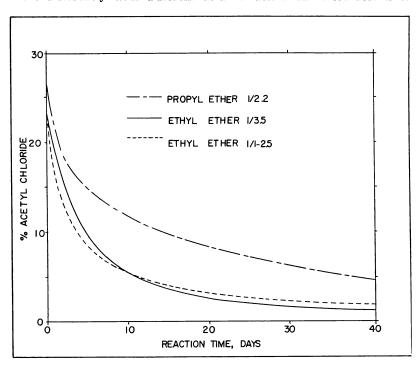
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### INTRODUCTION

Previous studies have indicated that acetyl chloride reacts with ethyl ether under suitable conditions to form ethyl acetate and ethyl chloride (3, 4, 5). However, no extensive study has been made of the rate or extent of this general reaction as applied to other

acid halides or ethers. If this reaction is found to be typical of other similar combinations of compounds, then a detailed study of the same should be of interest in interpreting the rate and order of similar reactions. Specifically different simple ethers might behave differently with the same acid halide. Likewise the same ether might react differently with different acid halides. From these results it



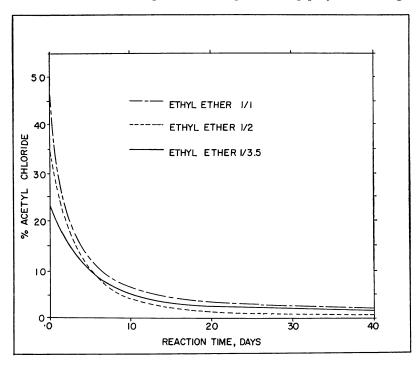
should be possible to draw conclusions regarding the reactivity and affinity of the related ethers or acid halides. Finally the use of mixed ethers, which theoretically should produce two distinct but related esters and alkyl halides, should provide the most significant evidence regarding the validity of the results obtained from the simple ethers:

This investigation was confined to a study of the reaction of acetyl chloride and three simple aliphatic ethers. All experiments were conducted under anhydrous conditions at a constant temperature of 25° C. Preliminary runs with the acid halide and the ether proceeded so slowly that the addition of a suitable catalyst was re-

quired. Anhydrous zinc chloride was selected as one of the best catalysts under the experimental conditions used (2).

### EXPERIMENTAL

The chemicals used in this series of experiments were purified acetyl chloride, ethyl ether, propyl ether, isopropyl ether, anhydrous zinc chloride, dioxane, and carbon tetrachloride. To control the temperature of the reaction a constant temperature bath, set at  $25^{\circ} \pm 0.2^{\circ}$  C. was employed. The reaction flasks consisted of 250 ml. iodine flasks with a length of 8 mm. glass tubing projected through



the side to the inside bottom perimeter of the flask so that aliquots could be withdrawn without disturbing the system.

It was necessary to devise a method of analysis for the solution so that the change in concentration of at least one of the components could be followed as the reaction proceeded. Since acetyl chloride was by far the most reactive component, it was decided to analyze quantitatively for the same. The following procedure was used. A convenient sized aliquot was withdrawn from the reaction flask, weighed on an analytical balance, dropped into 10 ml. of purified dioxane, and dissolved. An excess of water was added to the solution to hydrolyze any acetyl chloride that was present. This hydrolysis required

from ten to fifteen minutes to reach completion. The solution was then diluted to about 115 ml. with distilled water and titrated with standard sodium hydroxide using a pH meter to follow the course of the reaction and to establish the endpoint. Each mole of acetyl chloride present was hydrolyzed into one mole of aceticacid and one mole of hydrochloric acid. Each of these acids require an equivalent amount of sodium hydroxide for neutralization:

CH<sub>3</sub>COCl + H<sub>2</sub>O→CH<sub>3</sub>COOH + HCl → CH<sub>3</sub>COONa + NaCl + H<sub>2</sub>O

From this data the amount of the acid halide in the solution was calculated. The weight percent of acetyl chloride in the solution at the time of hydrolysis was recorded. The following graphs are the result of the weight percent of acetyl remaining in solution plotted against reaction time in days.

The initial experiments involved the use of acetyl chloride and ethyl ether in the presence of anhydrous zinc chloride. The effect of the catalyst, zinc chloride, on the speed of the reaction was first determined. It was shown that by holding the ratio of acetyl chloride to ether constant and by varying the amount of zinc chloride present, the speed but not the extent of the reaction was changed accordingly. After several determinations it was found that for every mole of acetyl chloride in the presence of 3.5 moles of ether, there should be 0.0107 moles of zinc chloride present to make the reaction proceed at a convenient rate and to still maintain a one phase system. This ratio of reactants to catalyst gave a first order reaction; whereas solutions with a higher catalyst ratio gave higher than first order reactions (1)-indicating the possibility of the formation of intermediates. However the extent of the reaction was not appreciably changed by increasing the catalyst/reactant ratio, but by increasing this ratio equilibrium was established much faster. In the above case the amount of remaining acetyl chloride was approximately 0.90 percent. However, it was very difficult to obtain accurate results when the acid halide content fell below 1 to 1.5 percent.

The effect of holding the acetyl chloride/catalyst ratio constant at 1/0.0107 and to vary the amount of ether present was next determined. The acetyl chloride/ether ratio was changed to 1/1.79 or just one half that of the preceding solution. This reaction proceeded much more rapidly than the preceding one, and gave a solution containing only about 0.70 percent acetyl chloride. The plot of this reaction follows closely to that of a typical second order reaction.

A solution of 1/1 acetyl chloride/ether ratio with the same catalyst ratio was tried. Its rate of reaction was much faster than either of the two preceding reactions showing that the speed of reaction is dependent upon the concentration of acetyl chloride present. This reaction follows quite closely a second order reaction.

Finally, an inert solvent, carbon tetrachloride, was mixed with

the ether in a 1/2.5 ether/carbon tetrachloride ratio. This solution was mixed with acetyl chloride to a 1/1-2.5 acetyl chloride/ether-carbon tetrachloride ratio with the same catalyst ratio. In the beginning this solution reacted comparably to the 1/3.5 acetyl chloride/ether solution, but in the later stages of the reaction it slowed up considerably.

Some work was done with propyl ether and acetyl chloride. Solutions of acetyl chloride and propyl ether at 1/5.4 and at 1/2.2 acetyl chloride/ether were tried. They both reacted much the same as the ethyl ether did except they reacted at a somewhat slower rate. Both of these solutions behaved as first order reactions.

Further work with isopropyl ether and acetyl chloride is being continued.

### DISCUSSION

It has been found that the concentration of the catalyst has a direct effect upon the speed but not necessarily the extent of this reaction. It is desirable to have enough catalyst present to cause the reaction to proceed at a comparatively rapid rate. But at the same time the concentration of the catalyst must be limited to the point where it will all dissolve in the solution, giving a one phase system. An excess of zinc chloride was converted to a very dense, viscous brown liquid which formed a second phase. Furthermore, a much more accurate analysis of the solution was obtained by using relatively small amounts of catalyst.

When the ratio of acetyl chloride to ether was kept low, about 1/3.5 or lower, the reaction proceeded as a typical first order reaction. That is, the speed of the reaction depends only upon the concentration of one component, which in this case is acetyl chloride. Here the ether is present in such a large excess that it is not a limiting factor. This is termed a pseudo first order reaction. However, when the acetyl chloride/ether ratio is more nearly equal, either 1/2 or 1/1, this same reaction has more nearly the characteristics of a typical second order reaction. The speed of this reaction is probably dependent both upon the concentrations of acetyl chloride and of ether. The 1/1 and the 1/2 reaction rates parallel one another quite closely in the initial stages of the reaction, but then the 1/2 slows down more in the later stages than does the 1/1. This may be due to a limiting effect because of a comparatively greater disappearance of acetyl chloride than of ether. This limiting effect is also shown in the comparison of a 1/3.5 acetyl chloride/ether solution to that of a 1/1-2.5 acetyl chloride/ether-carbon tetrachloride solution. In the initial stages of this reaction, the two proceeded at a nearly parallel rate; but later the latter reaction slowed up considerably. Again, this may be due to the disappearance of ether—it being the limiting component in this case. The inert carbon tetrachloride merely acted as a solvent.

The reaction between isopropyl ether and acetyl chloride proceeds at a relatively rapid rate even with very small amounts of catalyst present. In a matter of two days one solution proceeded from 28 percent to about 5 percent where it appeared to level off. This phase of the problem is still being studied.

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### THE FERRICINIUM CHLORIDE EQUILIBRIUM

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### INTRODUCTION

The discovery of a new class of compounds related to ferrocene (Dicyclopentadienyl iron) (1) has stimulated a large amount of interest and research (2). This study is concerned with the ferricinium ion, formed from ferrocene by the loss of one electron, rather than with ferrocene **per se**. This loss of an electron is analogous to the oxidation of the ferrous ion to the ferric ion.

$$(C_5H_5)_2Fe^+ + Cl^- = (C_5H_5)_2FeCl$$
  
 $Fe^{+2} = Fe^{+3} + e^-$ 

Both the ferric and ferricinium ions exhibit complex-formation with chloride ions.

$$(C_5\dot{H}_5)_2Fe^+ + Cl^- = (C_5H_5)_2FeCl$$
  
 $Fe^{+3} + Cl^- = FeCl^{+2}$   
 $FeCl^{+2} + Cl^- = FeCl_2^+$   
 $FeCl_2^+ + Cl^- = FeCl_3$ 

Ferrocene is an unusually stable compound in the sense that the two cyclopentadiene rings are firmly bound to the iron. Hence it is expected that a solution of ferricinium ions would not have a detectable concentration of ferric ion present, and the ferricinium chloride complex may be studied without interference from the other equilibria.

### EXPERIMENTAL

Excess ferrocene was oxidized by silver ion in a solution of silver perchlorate one molar in perchloric acid. The silver perchlorate solution was previously standardized with analytical reagent sodium chloride using Mohr's method. The resulting ferricinium ion solutions were found to be moderately unstable in neutral solution, and very unstable in basic solution.

To decrease the rate of decomposition of the solutions, nitrogen, purified by passing over hot copper turnings to remove oxygen, was bubbled through the reaction mixture. This had the added effect of mixing the insoluble ferrocene with the silver perchlorate solution. Complete oxidation required one hour when the reaction mixture was kept at  $0-5^{\circ}$  C.

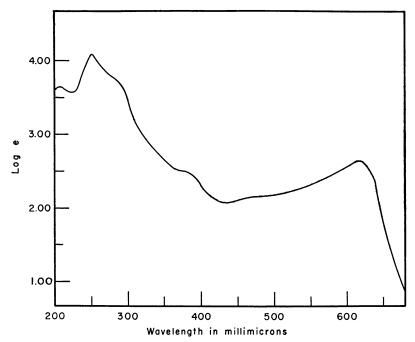


Figure 1. Log of molar extinction coefficients of ferricinium perchlorate solutions as a function of wavelength. Perchloric acid concentration 3.00 molar.

Precipitated silver and excess ferrocene were removed by filtering through a sintered glass cucible using a slight vacuum. The filtrate was diluted to the mark in a volumetric flask with conductivity water. Further dilutions were made from this stock solution.

A series of solutions was prepared, varying the ferricinium ion concentration from 4.11 x  $10^{-5}$  to 2.75 x  $10^{-3}$  molar, and the chloride

ion concentrations from 0 to 3.00 molar. The total acid concentration (hydrochloric plus perchloric) of all solutions was adjusted to 3.00 molar, giving a constant ionic strength of 3.00 molar.

A calibrated set of silica cells, 1.000 cm. in path length, was used and the optical densities recorded using a Beckman Model DU Spectrophotometer. A temperature of 20.0° C. was maintained in the cell compartment by circulating water through Beckman "thermospacers." Optical densities were recorded from 200 to 500 millimi-

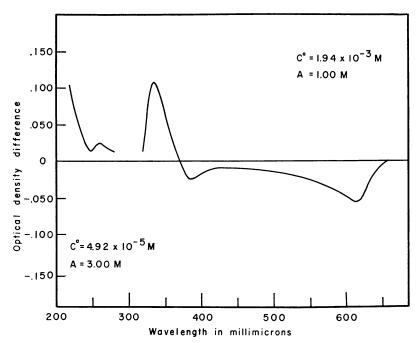


Figure 2. Optical density of ferricinium chloride solutions with reference to ferricinium perchlorate solutions at varying wavelengths.

Ionic strength 3.00 molar.

crons using a 1P28 multiplier photo-tube, and from 500 to 700 millimicrons using the standard red-sensitive phototube. Corrections were applied for absorption of both the perchlorate and chloride ions in the ultraviolet region.

Optical density readings as a function of time indicated that  $10^{-5}$  molar ferricinium ion solutions prepared in this way were stable for several hours while  $10^{-3}$  molar ferricinium solutions showed some decomposition within an hour.

### RESULTS AND CONCLUSIONS

Fig. 1 is a graph of the log of the extinction coefficient vs. wave-

length for ferricinium perchlorate solutions. Two maxima and two minima are found at 252 (log e=4.08), 620 (log e=2.64), 225 (log e=3.58) and 430 (log e=2.08) millimicrons.

Fig. 2 shows the optical density vs. wavelength of solutions of ferricinium perchlorate plus chloride ions with reference to solutions of the same initial ferricinium ion concentration From 210 to 370 millimicrons solutions with chloride ions absorb more light than do solutions with no chloride ions. The reverse is true from 370 to 650 millimicrons.

For a quantitative interpretation of the data three assumptions are made. (a) The solution obeys Beer's law, and the components have independent extinction coefficients. For cells of 1.000 cm. path length the equation is written:

$$D = eC + e' C'$$

where D is the optical density of the solution, e and e' are the extinction coefficients in liter/mol.-cm. respectively of the ferricinium ion and the complex, C and C' are the concentration in mol./liter of the ferricinium ion and complex. (b) An equilibrium constant can be written for the formation of the ferricinium chloride complex. The equation is written

$$K \, = \frac{C'}{CA}$$

where A is the chloride ion concentration in mol./liter. To a very good approximation A equals the initial concentration of chloride ions since A is much larger than C'. (c) An equation for the conservation of material can be written

$$C = C^{\circ} - C'$$

where C° is the initial concentration of the ferricinium ion.

Two sets of the three equations can be written for the same wavelength but different concentrations. The C, C' and e' terms are eliminated and the resulting equation solved for K in terms of measurable quantities. The subscripts 1 and 2 refer to two different chloride concentrations.

$$K \; = \; \frac{C_1{}^{\circ}A_1D_2 \; - \; C_2{}^{\circ}A_2D_1 \; + \; eC_1{}^{\circ}C_2{}^{\circ}(A_2 \; - \; A_1)}{A_1A_2(C_2{}^{\circ}D_1 \; - \; C_1{}^{\circ}D_2)}$$

Table 1 gives K calculated from representative experimental data at various wavelengths, ferricinium ion concentrations and chloride ion concentrations.

Table 2 shows K, averaged from twenty values, compared with literature values for ferric chloride equilibria (3). Ultraviolet data are not included in the average because of the large uncertainty in the corrections.

The error in the average K may be larger than the average deviations because of the time lag between preparing solutions and recording the optical densities. Certainly, however, the value for K

is of the same order of magnitude as the first and second constants of the ferric chloride equilibria and about fifty times as large as the third. Apparently then two cyclopentadienyl rings do not hinder the formation of a third coordinate bond to iron as much as do two chloride ions bound to ferric ion.

TABLE I
Experimental Data Use in Calculating K

Experimental Data Use in Calculating ix					
Wavelength millimicrons	$C_1^{\circ} = C_2^{\circ}$	$A_1$ and $A_2$	еС°	$D_1$ and $D_2$	K
	mol./liter	mol./liter			
230	$4.92 \times 10^{-5}$	0.300	0.196	0.236	4.9
		3.00		.259	
230	$6.56 \mathbf{x} 10^{-5}$	0.300	.266	.308	3.6
		3.00		.340	
560	$1.94 \times 10^{-3}$	0.100	.452	.444	2.4
		1.00		.423	
570	$2.33 \mathbf{x} 10^{-3}$	0.100	.605	.591	2.4
		1.00		.553	
580	$1.94 \times 10^{-3}$	0.100	.555	.545	2.2
		1.00		.518	
590	$2.33 \mathbf{x} 10^{-3}$	0.100	.748	.728	2.8
		1.00		.681	
600	$1.94 \times 10^{-3}$	0.100	.702	.689	2.3
		1.00		.654	
610	$2.33 \mathbf{x} 10^{-3}$	0.100	.965	.935	4.0
		1.00		.880	
620	$1.94 \times 10^{-3}$	0.100	.841	.827	2.4
		1.00		.790	
625	$2.33 \mathbf{x} 10^{3-}$	0.100	.955	.925	4.4
		1.00		.875	

TABLE II
Comparison of the Ferricinium Chloride and Ferric Chloride
Equilibrium Constants

Reaction	K	Temp.	Ionic Strength
	liter/mol.	°C.	mol,/liter
$Fe^{+3} + Cl^- = FeCl^{+2}$	$4.2 \pm 0.2$	26.7	1.00
	*6.4	20.0	3.00
$FeCl^{+2} + Cl^{-} = FeCl_{2}^{+}$	$1.3 \pm 0.4$	26.7	1.00
$FeCl_{2}^{+} + Cl^{-} = FeCl_{3}$	$0.04 \pm 0.02$	26.7	3.00
$(C_5H_5)_2Fe^+ + Cl^- = (C_5H_5)_2FeCl$	$2.9 \pm 0.7$	20.0	3.00
*Calculated for purposes of com	parison		

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# NEW METHOD FOR THE IMPROVEMENT OF THE QUALITY OF MUNICIPAL AND DOMESTIC WATER SUPPLIES

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### ABSTRACT

This paper presents the major results of field and laboratory studies on waters of six municipalities in North Dakota. The cities selected all had a problem of obtaining potable water free of excessive mineral content. An electro membrane diffusion process was employed in demineralizing these waters containing up to 4000 parts per million of total dissolved solids and excessive amounts of sodium, chloride, and sulfate ions which are not readily eliminated by standard methods. The operation was successful in reducing all waters treated to meet the United States Public Health Standards at an average power consumption of 18 kilowatt hours per 1000 gallons of product water.

### INTRODUCTION

Obtaining sufficient potable water is a serious problem in many communities and on many farms throughout the state. Temporary and permanent hardness can be removed by ordinary water softeners; but waters containing great amounts of sodium, sulfate, and chloride ions still present a serious technical and economic problem. The United States Public Health Standards for drinking water is not to exceed 1000 parts per million of total dissolved solids and not to exceed 250 parts per million for sulfates and chlorides (6) .Yet a number of municipal water sources in the state supply water reaching 4000 parts per million of total dissolved solids and 1000 parts per million of sulfate. Because of these concentrations it leaves the water certainly undesirable if not unfit for human consumption and general farm use.

The processes for producing fresh water from saline sources can be classified into two groups: (a) those that separate the water from the saline solution; and (b) those that separate the salt from the saline solution (5). The former include distillation, freezing, osmosis, and solvent extraction, while the latter include ion exchange and electrodialysis. Most of the proposed processes for the demineralization of saline waters depend on removing the water and leaving a concentrated brine. They therefore tend to have costs that are only secondarily a function of the salt concentration. Ion exchange takes up the salt, and it is eliminated with the acid and alkali regeneration waste streams. The electrodialysis process transfers the ions electrically through membranes that are semi-permeable with respect to particular ions. These last two processes have costs that are a

direct function of the total salt removed and to a lesser extent of the amount of water produced. They are particularly attractive for waters containing low original salt concentrations (2). Any process to be economically feasible must produce suitable water at a price which the average consumer can pay. The maximum delivered cost of water is frequently considered to be \$1.00 per 1000 gallons, although it may vary with local conditions; and according to recent cost estimates, water by the electrodialysis process will cost the consumer about \$.80 per 1000 gallons (7). This estimate considers the power cost to be 1½ cent per kilowatt hour and includes machine maintenance for a system supplying ½ million gallons per day. The electro-dialysis process is apparently the most economical in demineralizing brackish and saline waters for domestic consumption, and it is the process with which we are concerned in this paper.

#### DESCRIPTION OF EQUIPMENT

The electrodialysis process is extremely simple and is represented by the flow sheet in Figure 1. The essential part of the electrodialysis process is the membrane demineralizer. This consists of a series of 300 membranes with spacers separating them to allow a thin film of water to flow between the membranes. The odd numbered membranes remove cations and the alternate membranes anions, as illustrated in Figure 2. The cation membrane is an electrically conductive plastic sheet which permits the passage of only charged ions, such as sodium ion, under the influence of a d-c potential (185 Volts). The blockage or repulsion of anions is the result of the fixed negative groups evenly distributed within the insoluble matrix of the cation membrane. The anion membrane is similarly constructed but is selective for anions. These membranes are patented and chemically similar to ion exchange materials i. e., they contain a high concentration of an ion bound to a water-insoluble structure, and they contain a high volume fraction of water (2). The physical chemist describes them as "solid polyelectrolytes consisting of a hvdrocarbon cross-linked skeleton to which polar groups are attached" (1). The multi-cell system shown in Figure 2 indicates that each cell is composed of an anion membrane and a cation membrane suitably separated from each other by spacers to permit the passage of the saline water. The membranes alternate with regard to sign and the cells are electrically in series between a single set of electrodes. When a d-c voltage is impressed across the terminals, the resulting migration of the ionic constituents results of an increase in salt concentration in one-half the flow passages and a decrease in a multitude of highly ionized constituents from non-ionized or slightthe others. This basic multi-cell system can be utilized to separate ly ionized constituents (5). In the case illustrated, the ionized constituent is sodium chloride and water is the slightly ionized constituent. The power required is direct function of the separation maed and thus depends upon both the original and final ionic con-

FIGURE 1.

#### FLOW SHEET

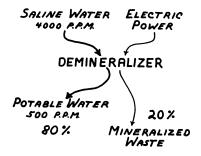


FIGURE 2.

MEMBRANE ARRANGEMENT

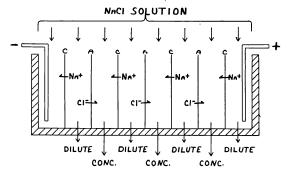
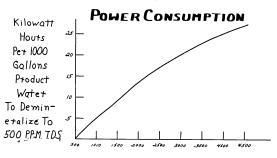


FIGURE 3



Total Disolved Solids
In Source Water - PPM.

centration. The other major cost element is membrane replacement.

The equipment as operated this summer consisted of a single membrane stack as described and illustrated, together with suitable pipes and controls to make a functioning unit. The concentration of the product water is automatically controlled and can be adjusted to any desired concentration. The membrane stack has a capacity for 40%-50% demineralization in one passage at a rate of 20 g. p. m. of water fed to the diluting cells. Higher demineralizations is achieved either by passing the product to additional stacks in series, or recirculating it through the same stack until the desired purity is achieved. For the purpose of economy and compactness, the later procedure was followed in this investigation.

#### **EXPERIMENTAL**

After a period of preliminary study at the University of North Dakota, first on synthetic and second on water hauled in from Grafton and Devils Lake, North Dakota, the equipment, as previously described, together with suitable tanks and test equipment was mounted on a five ton truck. The equipment was then transported to selected stations inthe state.

Table 1 presents the pertinent date for each of the major tests. The most highly mineralized water encountered was well water at Grafton running at 4,080 p.p.m. It also had the highest sulfate content (985 p.p.m.) of any water studied and the second hihgest concentration of hardness and alkalinity. The power requirement to produce a potable water of 540 p.p.m. total dissolved solids was 23.5 kilowatt hours per 1000 gallons. Lakota had the lowest total dissolved solids (1531 p.p.m.) but had the highest hardness at 685 p.p.m. and the third highest sulfate (774 p.p.m.). Potable water of 508 p.p.m. total dissolved solids was produced at 16.1 kilowatt hours per 1000 gallons.

No precise temperature control was utilized, but the range of operating temperature was 50°F (Lakota) to 70°F, and in the opinion of the operators this change had little affect on the operation of the machine with the possible exception of Lakota. The higher the temperature, the greater the efficiency of the machine, and therefore the least amount of power consumption. Normally, therefore, an increase in water temperature is desirable; but the maximum safe feed water temperature for the membrane stack is 120°F.

The power consumption per 1000 gallons of product water is a function not only of the initial and final ion concentration of the water, but also of the reduction ratio. The actual consumption can vary widely because of the flexibility of the process as shown in Table 1. No satisfactory theoretical relation has been worked out between the mineral content and the power consumption, however Figure 3 shows the empirical relation as determined by these six

# TABLE 1

	Gra	Grafton	De	Devils	Bowbells	ells	Stanley	ıley	Ellen	Ellendale	Lakota	ota
a—initial analysis			La	Lake								
b—final analysis	Ø	р	ø	q	๙	р	Ø	р	ฒ	р	ત્ય	q
Total Dissolved Solids p.p.m.	4080	540	4066	512	2460	470	1820	450	2600	472	1531	208
Hardness p.p.m. as CaCO <sub>3</sub>	219	20	20	∞	32	2	92	18	09	6	685	202
Calcium p.p.m. as CaCO <sub>3</sub>	119	11	29	4	17	က	58	11	32	4	460	124
Magnesium p.p.m. as CaCO <sub>3</sub>	100	∞	21	က	15	2	34	7	28	2	225	80
Alkalinity (HCO <sub>3</sub> ) p.p.m. as CaCO <sub>3</sub>	730	160	687	148	1425	369	726	207	503	145	385	203
Iron p.p.m.	_	0	0	0	0	0	0	0	0	0		
Sulfate p.p.m.	985	117	823	175	80	0	684	181	159	33	774	200
Chloride p.p.m.	187	12	120	6	42	0	0	0	173	20	9	
Hď		İ	8.2		7.9	7.7	8.0	8.1	8.2	7.1	8.2	7.9
Kilowatt hours required per												
1000 gallons of product water	2	23.5	2	21.5	16.3	ယ်	14.4	<del>4</del> :	15.9	o:	16	16.1

tests. This relation has an empirical formula of:

$$y = a(x - b)^c$$
 or  $y = 0.203(x - 500) 0.575$ 

#### Where:

- a. is a conersion factor, the units of which are kilowatt hours per 1000 galons of product water per unit of reduction in total dissolved solids; and it is probably a function of the operating temperature.
- b. is the arbitrarily determined lower limit of total dissolved solids in parts per million.
- c. is interpreted as being a function of the ratio between the mono-valent and di-valent ions.
- x = Initial total dissolved solids in p.p.m.
- y = Total kilowatt hours required to produce 1000 gallons of potable water with 500 p.p.m. total dissolved solids.

#### CONCLUSION

Since no satisfactory means has previously been developed for demineralizing saline and brackish waters to meet the economic requirements of this region, this investigation indicates that the electrodialysis process using the patented semi-permeabel membrane is able to convert water from this region into potable water meeting the United States Public Health Standards at a cost that the average community can afford.

#### ACKNOWLEDGEMENTS

I wish to thank Dr. Alex C. Burr and the North Dakota Research foundation for permitting me to have access to the files of data on experimental work. I am also indebted to Prof. A. M. Cooley of The Chemical Engineering Department at the University of North Dakota who supervised the research. Thanks is also due to Pat Markovic who accompanied me in the field and to Jim Innes and George MacKenzie who helped in preparing the machine for field work.

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#### SLIDES WITHOUT A DARKROOM

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#### ABSTRACT

The availability of adequate darkroom facilities or specialized photographic equipment often restricts the use of lantern slides as a means of illustration and presentation of data. Both tabulated and graphic material can be made into slides non-photographically by drawing or typing characters on "Radio-Mats\*;" drawings can also be made on transulcent plastic sheet\*\*. Material prepared in this manner masked and sandwiched between slide cover glass makes up into presentable slides.

Photographic slides can be prepared without using a darkroom by taking advantage of commercial color-film processing. Photographs of laboratory apparatus, specimens, geologic and geographic scenes need no comment here. However, one can take advantage of color-film in preparing slides by photographing illustrations made by means of a flannel-board and titling letters\*\*\*. Any 35 mm. camera capable of focussing down to 3 ft. can be used since most copy will be taken from a distance of about 4 ft. Illustrations that lend themselves well to the technique are the scatter-graph, curves, circlegraph and bar-graphs. In order to obtain full advantage of color one should use harmonious but contrasting vivid color for background, bars, lines and lettering. Examples of each of the above are presented.

- \*"RADIO-MAT Slide" material may be obtained from Radio-Mat Slide Co., Daytona Beach, Fla.
- \*\*Material such as "TUFFILM," matt, heavy weight no. 197-300 may be obtained from M. Grumbacher, N. Y., through a stationery store.
- \*\*\*Suitable titling letters are manufactured by Mitten's Display Letters, Redlands, California, and are obtainable through photographic dealers.

#### PHYSIOLOGY OF COLD

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In the last decade arctic areas have become increasingly important because of their strategic location for military operations, and because they contain unexploited natural resources. The impact of the arctic climate on man has raised some interesting problems for the physiologist, especially with regard to the development of cold adaptation, and the effects of long-time cold exposure on bodily functions. From the practical standpoint, the study of the effects of cold on man have led to the development of more effective clothing, and new methods for preventing and treating frostbite and other cold injuries.

As every North Dakotan knows, when man and animals are confronted by winter, there are but two choices: migrate to a more temperate climate, or adapt to the cold. Adaptation is not entirely a matter of avoiding actual freezing, but of being comfortable and able to do work at low air temperatures. Even a slight lowering of internal body temperature has a profound effect on physiological processes essential to life. Should the internal temperature drop from 98.6° F to 90°, for example, there will be slowing of essential chemical reactions in the nervous system, and intelligent and coordinated activities become impossible. Long before this happens, however, defense responses to cold have been set off, and only whn these have been overwhelmed does a fall in deep body temperature occur.

Temperature regulation is mediated through a controlling center in the brain called the hypothalamus. This center receives impulses from cold receptors in the skin, and after suitable integration, relays stimuli to the heat producing and conserving mechanisms. For instance, when a man is subjected to cold, he makes involuntary movements of his muscles, a type of exercise called shivering, which is a method of producing heat. The heat thus generated can more than double the normal production. This heat may be further augmented by voluntary arm swinging and other activity by the cold victim.

Heat conservation is another defensive action against cold. In man, the absence of fur is a distinct handicap, but he nevertheless can use his skin as insulation. Nerves to the superficial blood vessels cause narrowing of these channels, and less warm blood enters the skin where its heat could be lost to the air. Some exposed areas of the body, however, particularly, the hands and feet, are more likely to be frostbitten than others, and blood flow to these parts must

be kept up lest the parts be rendered numbed and useless by the cold.

Many observations and experiments have shown that long-time exposure to cold increases the efficieny of the heat producing and conserving functions. For example, an Eskimo was unperturbed when placed in a tub of cold water at 50° F, whereas non-adapted subjects were miserably chilled, and had to give in to the cold long before the experiments were completed. Another example of cold adaptation, or perhaps it could be simply a matter of learning to get along in a cold climate, was disclosed in a medical report on frostbite causualties in the recent Korean War. The frostbite incidence in soldiers from southern parts of the United States was four times greater than in those reared in the northern zones. Many other observations indicate that man has the ability to adapt to cold, but certainly to a less extent than do animals under the same circumstances.

Some of the more remarkable adaptations occur in arctic animals, particularly in sea-dwelling mammals such as the porpoise and whale. These animals are surrounded by cold sea water at all times, yet manage to keep the body temperature constant at the same level as man's. The swimming mammals are particularly susceptible to high heat loss from flippers and tail, which present large thin surfaces from which heat can escape readily. However, this is avoided to a large extent by a peculiar arrangement of the blood vessels to form a heat-exchange system. Gross anatomical and microscopic studies of the tail of the porpoise revealed that the arteries supplying the tail-fin are surrounded by a cluster of veins. Warm blood in the artery gives up its heat to the blood in the veins, which is returning to the body. This mechanism prevents needless loss of heat, without depriving the highly active tail of the oxygen and nutrients carried by the blood.

Heat conservation by insulation is another means of keeping the body temperature constant in a cold environment. Man can adjust his clothing to meet temperature changes, and animals do the same, but on a much longer time basis. Growth of hair coat in fall, and its shedding in spring are common occurrences seen in domestic and farm animals. Careful studies of the fur insulation of arctic animals have been made by scientists in an effort design more effective clothing for military operations in cold climates. These studies have indicated that fur is highly effective in land dwelling forms, and that a thick layer of subcutaneous fat is the primary insulation of aquatic animals, such as the seal and whale.

Insulation values of various kinds and thicknesses of clothing have been measured and a standard of insulation, the "clo" unit, has been adopted. As an example, the average-weight woolen business suit has a value of one clo unit. The arctic white fox, caribou,

and wolf have fur insulation equivalent to 8 or 10 clo units, or suits of clothing, worn at one time!

Long-time exposure to cold develops a greater capacity to generate heat, as well as insulation layers to conserve it. Temporary means of increasing heat production are by shivering, and by voluntary movements such as swinging the arms and stamping the feet to stimulate blood flow and make muscles generate heat. An animal continuously in a cold environment, however, has its heat production stimulated by the thyroid hormone, which is produced in larger amounts on long-time cold exposure.

In order to produce more heat by any means, greater quantities of food must be taken into the body, where it can be oxidized to heat and energy. Again, this is a topic of military importance, and nutritionists are endeavoring to find the most suitable diet for cold climates. Up to the present, it has been found that army personnel prefer about the same types of food, but eat twice as much when on maneuvers in Alaska as compared with the same type of activity in Florida.

Most animals, as well as man, have poorly developed temperature regulating mechanism at birth. Newborn dogs, for example, behave as inanimate objects when placed in warm or cold environments; that is, the body takes on the temperature of the surrounding air. As the animal ages, it becomes more resistant to heat or cold, until at the age of 3 or 4 weeks it regulates the body temperature quite efficiently. In man, the same development appears to take place, except that it takes considerably longer for complete temperature regulation to be acquired. The details of the development of temperature regulation have been studied in the Department of Physiology and Pharmacology at the University of North Dakota during the past several years. In the dog, regulation in a cold environment of about 4 degrees Centigrade is practically non-existant during the first 5 days after birth. At this age animals show a steady drop in temperature in the cold, much like the decline shown by a jar of saline solution of the same weight as the animal. As development proceeds, the temperature drops less, and at the age of 3 weeks most dogs studied managed to keep body temperature constant in the cold room. The mechanisms involved in the developmental process are many, and to date only a few have been studied. Most noticeable was the observation that heat generation becomes more effective through the increase in duration and strength of shivering movements.

The rise in heat production in dogs at various ages was measured by recording oxygen consumption, and converting the values to calories per square meter of body surface per hour. In Table 1 the gradual development of the ability to resist cold can be seen to coincide with the rise in heat production. In the cold stress used the animal had to double its normal heat production in order to

maintain its temperature constant at about 37° C. As can be seen in the table, dogs under 2 weeks of age failed to do this, and, therefore, body temperature dropped. Animals slightly older than this, between 3 and 4 weeks of age, were able to maintain a constant temperature. The cold resistance acquired at this age most likely results from greater heat production in the muscles and other organs such as the liver. However, other factors, including increase in fur thickness, and shifting of blood away from the skin, probably become important in conserving heat as the animal matures.

Experimental data on extremes of body temperature experienced by human subjects are, of course, lacking, but a few case histories of accidental cooling have been recorded in medical journals. The most spectacular of these is the report of a woman who recovered from a deep body temperature of 60° F, but suffered severe frost-bite and partial loss of hands and feet. The details of this case can be found in the medical journal report listed below. In general, it was found that consciousness returned at about 85 degrees, and that heart and blood pressure responses were approaching normal at about 95 degrees. From this and other observations it is apparent that man and the warm-blooded animals are limited to a narrow zone in the internal environment, but, by various methods of adaptation, can withstand vast extremes of external environments.

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TABLE I

Changes in deep body temperature and heat production in dogs of various ages subjected to air temperature of  $4^{\circ}$  C for one hour.

Age in days	Drop in body	Per cent increase in
	temp.	heat production
1 -5	13° C	18
6 -10	8	40
11-15	4	67
16-20	<b>2</b>	79
21-25	2	82

### INFLUENCE OF MUSCLE TENSION ON EXOSTOSES OF LATHYRIC RATS\*

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#### ABSTRACT

It has long been known that rats fed a diet of which one-half is meal of the sweet pea Lathyrus odoratus develop exostoses and other abnormalities of the skeleton. The present investigators have shown that exostoses of lathyric rats are formed from tissues of the inner layer of only those areas of the periosteum which serve for attachment of muscles. That observation suggests that stimulation to exostosis formation by a sweet pea factor is effective only in the presence of stimulation by a second factor, possibly muscle tension.

To test the second factor hypothesis, eleven adult rats were subjected to transection of muscles attached to areas of periosteum which give origin to two exostoses, the iliacus-psoas exostosis and the pectineus-adductor longus exostosis. The iliacus, psoas, pectineus and adductor longus muscles of the left posterior appendage were sectioned while no muscles of the right posterior appendage were sectioned. Ten of the animals subjected to muscle section were placed on the sweet pea diet and killed after 6, 12, 13 and 14 weeks on the diet. The remaining animal was fed a regular laboratory diet and killed after 21 weeks.

Though the control rat remained free of exostoses, exostoses and other abnormalities of the skeleton appeared in all animals fed the Lathyrus diet. Exostoses of those animals were typical in size and shape for all parts of the skeleton excepting the left femur. The exostoses at the insertion of the transected muscles failed to appear. This experiment, therefore, demonstrates that the sweet pea factor alone cannot produce exostoses but that it can do so in the presence of tension of intact, functional muscles.

\*This investigation was supported by research grant C-2153(C4) from the National Cancer Institute of the National Institutes of Health, United States Public Health Service.

### THE EFFECT OF EXERCISE ON LATHYRUS INDUCED EXOSTOSES\*

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#### ABSTRACT

Because most of the skeletal deformities of adult rats fed a diet containing large amounts of **Lathyrus odoratus** are related to the areas of muscle attachment, and because the primary symptom in human lathyrism is spastic paralysis, muscle pull would seem to be an intimate factor in exostosis development. That muscle pull is essential to the formation of exostoses in lathyric rats was shown by experimental muscle section which prevented their development.

If one postulates that the appearance and size of the exostosis is related to the strength and frequency of action of the muscle, then one would expect larger exostoses to appear at the attachment of the larger and most useful muscles of the rat. This seems to be the case since exostoses do not appear at all muscle attachments. Also, if this hypothesis is correct, a more active rat should produce larger exostoses than an inactive rat.

An experiment was therefore undertaken in which animals fed **Lathyrus odoratus** were separated into exercise and non-exercise groups. The exercise group was forced to exercise for one-half hour daily in a cage which revolved at a speed which made the animals walk rapidly but not run. After 3, 6 and 12 weeks of experimentation the animals were killed, autopsied and fixed in 10% formalin. The skeletons were then cleaned of muscles and the size and time of appearance of exostoses was studied.

When the exostoses of animals which were forced to exercise were compared to exostoses of animals not forced to exercise, no real difference was noted either in size or in time of first appearance. It therefore appears that a minimal stimulus is required in order for exostoses to appear, but no difference in size or rate of growth is effected by above minimal stimulation.

\*This investigation was supported by research grant C-2153(54) from the National Cancer Institute of the National Institutes of Health, United States Public Health Service.

#### CONGENITAL FELINE CEREBELLAR MALFORMATION

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#### ABSTRACT

A three week old female kitten, with a diagnosis of cerebellar ataxia, was observed, photographed, and tested for 14 weeks and then sacrificed. Serial sections of the cerebellum and other portions of the brain were strained alternately according to the techniques of Nissl and Weil. The parents and littermates were considered to be normal.

Neurologically there was ataxia, tremor, and asynergia as evidenced by increased amplitude and frequency of oscillations during voluntary activity or when conscious attempt was made to fix or

maintain position of the head. There was exaggeration of positive supporting reactions which was most evident in the hind limbs. When sitting the forelimbs were extended and abducted and the hindlimbs flexed and adducted, forming a tripod, which was the best stable position the animal could assume.

There was a normal labyrinthine righting reflex and no nystagmus (spontaneous or following quick eye movements). Visual, auditory, and cutaneous sensibilities were judged normal.

The brain and cord were grossly normal with the exception of the cerebellar hemispheres which were markedly reduced in size, and the vermis, which was moderately reduced in size.

Histologically the Purkinje cells were observed scattered throughout the molecular layer. They were polymorphic, stained with varying intensities, had variations in amount of Nissl substance, and many had eccentric nuclei. There was extreme variability in the thickness of the granular layer. The malformation of the hemispheric extensions of the vermian lobules was especially severe in the folium and tuber vermis.

An attempt was made to correlate the physiological deficit with the cerebellar malformation.

### METABOLIC CHANGES IN DOGS DURING DEVELOPMENT OF TEMPERATURE REGULATION

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#### ABSTRACT

Adult mamals (homoiotherms in contrast to cold-blooded animals (poikilotherms) are able to maintain a constant body temperature in the face of a cold environment, such as a North Dakota winter. However, most newborn mammals, including pups, are poikilothermic and must develop temperture regulation postnatally. This development revolves around two main factors, heat production (chemical) and heat conservation (physical). It is of basic physiological interest to determine the quantitative aspects of temperature regulation; therefore, experiments were undertaken to determine the heat production of pups at various ages.

The  $0_2$  consumption was measured in a closed metabolism chamber. The body temperatures and weights were also recorded. From this data the metabolic rates and heat losses were calculated.

All 5 age groups studied showed no rise in metabolic rate during the one hour exposure to 30°C. However, it was found that

basal heat production was higher with increasing age. In 1-5 day old dogs the metabolic rate averaged 34.6 Cal/m²/hr. and in 21-25 day old dogs the rate averaged 44.6 cal/m²/hr. The rectal temperatures remained constant for all age groups when the air temperature was  $30^{\circ}$ c.

At air temperatures of 22° to 24° C. the metabolic rates of pups 1-5 days old increased and rectal temperatures fell a few degrees. In the older dogs the metabolic rates rose progressively less at this temperature until finally in the oldest group, 21-25 days, there was no rise during a one-hour exposure. There was essentially no fall in rectal temperature in the older animals.

In the cold room (about 5°C.) the metabolic rate for dogs 1-5 days old increased to 40 Cal/m²/hr., and the rectal temperature fell several degrees. Similar changes occurred in the pups of 6-10 and 11-15 days old except the metabolic rates rose higher and the rectal temperatures did not fall as much. In 16-20 and 21-25 day old animals the metabolic rate rose to a much higher level and the rectal temperatures fell very little. Pups of this age were considered to be homoiothermic.

(This project was supported by U. S. Public Health Service Grant RG-H-4580.)

#### HISTOCHEMICAL OBSERVATIONS ON METAPLASTIC EPITHELIUM OF THE RAT UTERUS FOLLOWING ESTROGEN TREATMENT\*

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#### ABSTRACT

Glycogen, cytoplasmic ribonucleic acid and alkaline phosophatase were studied in metaplastic epithelium of the rat uterus following treatment with estradiol dipropionate.

To demonstrate glycogen, the periodic acid-leucofuchsin technic following the procedure of McManus was used. Control sections were incubated for an hour in 1% malt diastase buffered at pH 6.8. In order to study cytoplasmic ribonucleic acid, sections were stained with toluidine blue in a citrate solution buffered at pH 4.5 for one hour at 37° C. Control sections were incubated with a solution of crystalline ribonuclease (1 mg. per 100 cc. buffered at pH 6.8) for one hour at 56° C. To demonstrate alkaline phosphatase activity, sections were treated with 1% sodium glycerophosphate substrate for two hours at 37% C and treated according to the technic of Gomori.

In the metaplastic epithelium, small numbers of glycogen granu-

les were observed in the epithelial cells. A marked amount of cytoplasmasmic basophilic material was observed in the basal and intermediate cells of the metaplastic epithelium, while the superficial cells contained fine granules which were not numerous. Deposits of colbalt sulphide, indicating the presence of alkaline phosphatase, was observed only in the intermediate and superficial layers of the stratified metaplastic epithelium.

The significance of these materials in metaplastic epithelium is not definitely known. However, glycogen may be a source of readily utilizable energy for mitotic activity, while cytoplasmic ribonucleic acid may be correlated with protein synthesis and alkaline phosphatase may be related with fibrous protein synthesis.

\*Supported by a grant from the National Vitamin Foundation.

# THE ELECTROPHORETIC SERUM PROTEIN PATTERN OF RATS FOLLOWING ADMINISTRATION OF BROMOBENZENE

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#### **ABSTRACT**

Liver necrosis was produced in 29 male Sprague-Dawley rats by the administration of bromobenzene in corn oil (Proc. Exp. Biol. Med. 79, 196, 1952). Blood was removed at 2, 6, 12, 24, 48, 72 and 96 hours following the administration of the hepatocellular toxin for the analysis of serum protein (Micro-Kjeldahl) and electrophoretic fractionation (Perkin-Elmer Tiselius Model). Twenty-two control animals received an injection of corn oil. The area data from the electrophoretic pattern were expressed as grams % protein. Initially the albumin and  $\alpha_1$  globulin increased to a maximum at 6 hours, then decreased to a minimum at 18 hours with the albumin fraction returning to its initial value rapidly. However the  $\alpha_1$  globulin did not return to its initial value but followed the trend of the control curve. The  $\beta$  globulin area decreased slowly until the 18 hour interval then increased to its initial value at the 72 hour interval. The  $\alpha_2$  and  $\gamma$  globulin increased at the 18 hour and 6 hour period respectively with the ag globulins decreasing rapidly initially then slowly to almost the initial values, while the  $\gamma$  globulin remained elevated for 12 hours then decreased to below the initial value. An unknown fraction appeared in both the control and injured animals, but its appearance was delayed initially in the animal with necrosis; then this fraction increased slowly to a level above the initial and control values.

The mobility of the electrophoretic protein fractions was calculated. The mobility of the albumin,  $\alpha_2$  and the unknown fraction was higher than the control value in all the time periods except in the later time intervals when the albumin and  $\alpha_1$  dropped below the control value. The mobility of the  $\alpha_2$  globulin was lower initially then increased above the control level and remained elevated until the 50 hour interval at which time it fell below the control level. The  $\gamma$  and  $\beta$  globulins mobilities did not change initially but both fractions became elevated at the 12 and 18 hour interval respectively for the duration of the time period.

#### EFFECT OF HYROCARBONS ON THE URINARY PAR-TITION OF S<sup>35</sup> FOLLOWING THE ADMINISTRATION OF S<sup>35</sup> METHIONINE

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#### ABSTRACT

Various organic solvents capable of producing liver necrosis disrupt fundamental processes in the metabolism of sulfur-containing compounds by the hepatic cell. Previous data obtained in this laboratory demonstrated that the administration of bromobenzene and carbon tetrachloride caused depetion of free cystine in the liver of the rat. It was postulated that the level of free cystine in the cell may be the limiting factor in the production of necrosis. It is known that the liver detoxifies bromobenzene as a mercapturic acid. The study reported here was undertaken to gain further information which would perhaps indicate the general nature of the mechanisms involved in the *in vivo* detoxification of other organic solvents.

Equimolar corn oil solutions of bromobenzene, CCl<sub>1</sub>, HCCl<sub>3</sub>, and benzene were administered intrapertitoneally immediately following intraperitoneal injection of 15 μc of S<sup>35</sup> L-methionine to male Sprague-Dawley rats (250 g.) which had been fasted 24 hours before injection. A control group was injected with corn oil. Each rat was placed in an individual metabolism cage which allowed the separation of urine and feces. The thirty-hour urine samples were collected and made phosphate free. Total S<sup>35</sup> and inorganic S<sup>35</sup> of urine aliquots were determined. The organic sulfur was defined as the difference of these two fractions. There was observed in the urine of the control animals 63% of S<sup>35</sup> in the inorganic fraction. The administration of bromobenzene and benzene resulted in 85% and 66% respectively, of the S<sup>35</sup> activity being found in the organic fraction. CCl<sub>1</sub> and CHCl<sub>3</sub> ad-

ministration showed just the opposite effect, with 74% and 72% of the S<sup>35</sup> found in the inorganic fraction. The mechanism of the decreasing content of free cysteine in the liver following administration of bromobenzene is apparently different from that following insection of CCl<sub>4</sub> and CHCl<sub>5</sub>. The concentration of cysteine in the liver cell is very important and may be involved in the protection from various hepatotoxins which are involved in the production of liver necrosis

### THE ROLE OF THE SULFUR-CONTAINING AMINO ACIDS IN WOUND HEALING

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#### ABSTRACT

It has been shown in several ways by a large number of workers that the sulfur-containing amino acids methionine and cystine have an accelerating effect on the healing rate of wounds. However, the specific chemical mechanisms involved are not known.

By pre-labeling predominantly the more labile body proteins by injecting S<sup>35</sup> -L-methionine, inflicting a 3 cm. circular skin wound on the back of white rats, and then performing chemical and radio-activity analyses leading to the calculations of the "specific activity" (ratio of radioactivity/concentration of amino acid sulfur) of cystine + cysteine (hereafter referred to as CYSTINE) and methionine in the various tissues of control and wounded animals, it has been possible to gain insight into the breakdown, redistribution and incorporation of the sulfur-containing amino acids following wounding of white rats on a zero per cent protein diet.

It was found that, immediately following injury, as compared with similar values for control animals, there was a decrease in the methionine and CYSTINE specific activity in wounded rats' muscle and skin tissues, and a concomitant rise in that for kidney and both protein and non-protein fractions of liver. Previous findings indicate a rapid rise in wound methionine in the early stages of regeneration and a later, greater, prolonged rise in CYSTINE; that almost all of the administered L-methionine-S<sup>35</sup> incorporated into the wound exists as protein-bound methionine or CYSTINE is indicated by comparatively high specific activities for these materials at this site. A small fraction of the S<sup>35</sup> also was present in the wound as mucopolysac-

charide sulfate and as non-protein amino acids and peptides. Considering these observations and the fact that the specific activity of both protein and non-protein bound blood plasma methionine and CYSTINE was higher in wounded than control animals, it is postulated that rats on a non-protein diet respond to the demand of regenerating wound tissues for the structural materials methionine and CYSTINE by breaking down their more labile body proteins and mobilizing the sulfur-containing amino acids either directly to the wound, or, in the case of methionine, partially to the liver and kidney as well, where it is converted into CYSTINE, which then is transported to the wound tissue and incorporated into proteins. (This work was supported in part by a grant from the North Dakota Cancer Society).

### THE PINWORM, Enterobius vermicularis (Linnaeus, 1758), IN CHILDREN OF A NORTH DAKOTA VILLAGE

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and

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#### ABSTRACT

Examination of 203 children, ages 6 to 8, of a total of 237 in the first three grades in a North Dakota village (population 1500) school, revealed 41 percent positive for pinworm. A modified Graham (1941) method utilizing Scotch tape, adhesive side out, around a glass stirring rod, was used for all perianal examinations. Precautions were used to reduce contamination to a minimum.

More boys (47 percent) than girls (36 percent) were found to be positive for the parasite. Forty percent of the first grade, 46 percent of the second, and 38 percent of the third exhibited ova. Thirty-eight percent of the rural children and 44 percent of the urban children were infected according to the examination.

Instances were siblings were involved revealed 9 sets both negative, 10 sets both positive and 17 sets with one positive and one negative.

The high incidence occurred at a time in early May, 1956 when weather conditions had been severe enough to hamper outside play. Graham, C. F. 1941 A device for the diagnosis of **E. vermicularis.** Amer. Jour. Trop. Med. 21: 159-161.

# STUDIES ON THE LIFE CYCLE OF CRYPTOCOTYLE CONCAVUM FROM THE COMMON SUCKER AND EXPERIMENTALLY IN THE CHICK

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#### ABSTRACT

During the examination of fish from Turtle River, North Dakota in 1956, 11 of 29 common suckers, Catostomus c. commersonnii were found infected with 43(6 to 184) specimens of a metacercaria new for North America. None of the other fish examined (Semotilus a. atromaculatus, Notropis cornutus frontalis, Notropis d. dorsalis, Esox lucius, Bolesoma n. niqrum, Hadropterus maculatus) were found infected with this parasite. The worms were encysted in the musculature of the fish and were recovered by the pepsin HCl digest technique.

The larva is enclosed in an oval cyst of parasite origin 267(166-405) by  $162(135-216)\mu$  which is enclosed in a melanated host cyst. The live metacercaria is wide and flat 365(302-450) by  $267(211-345)\mu$  with oral sucker  $36\mu$ , pre-pharynx  $6\mu$ , pharynx 36 by  $25\mu$  esophagus  $34\mu$  and ventral sucker  $34\mu$ . The cuticle is minutely spined, and the excretory bladder is Y-shaped. There are two very prominent lateral tubules which terminate near the oral sucker.

Four chicks, 1-3 days old were given 40-290 metacercariae each per os; 3-13 adult worms were recovered from the intestine of three chicks in 3-4 days. The adult trematode, fixed in hot Bouin's solution, stained and mounted, is wide and flat, minutely spined, and is 486 (418-585) by 266  $(240-313)\mu$ . Oral sucker  $37(34-39)\mu$ ; pre-pharynx 12  $(5-20)\mu$ ; pharynx 30(24-36) by  $28(24-31)\mu$ ; esophagus 46(36-61); intestinal caeca slender  $(7-12\mu)$  and extending to posterior end of body. Ventral sucker  $50\mu$ , situated in mid-line, gonotyl  $17(12-22)\mu$  on anterior edge. Testes oval 60(36-73) by  $98(78-115)\mu$  and situated opposite each other in posterior part of body, their median border separated by the stem of the excretory vesicle; a structure containing sperm and thought to be the seminal recepticle is elongate and extends transversely just anterior to the testes; no cirrus pouch present; seminal vesicle sausage shaped, extending transversely just on left of ventral sucker. Ovary round 51(49-54) \( \mu, \) just anterior to left testis. Vitellaria lateral and composed of medium sized follicles  $(9-12\mu)$  which extend to about halfway between ventral sucker and fork of intestine with transverse sheets meeting just anterior to ventral sucker. Uterus relatively short and forming an S-shaped loop which lies in mid-line between ovary and gonotyl. There are 13 to 24 oval, golden yellow eggs 32 by 18μ in utero which have definite shoulders. Excretory vesicle Y-shaped with wide stem and relatively short, slender branches; excretory pore terminal. The two conspicuous lateral tubules are still present in the adult.

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### BALLISTOCARDIOGRAPH STUDIES ON THE WELL TRAINED ATHLETE\*

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#### **ABSTRACT**

The ballistocardioraph is a comparatively new instrument used in the study of the cardiovascular system. Among the types of analysis for which this apparatus has been used is the estimation of the cardiac stroke volume.

The purpose of this project was to determine if the ballistocardiograph can be used to estimate cardiac stroke volume after exercise and to ascertain whether or not physically conditioned athletes show a greater stroke volume both before and after exercises than the non-athletic person.

To obtain supplementary data, studies were made of the electrocardiorgam, vital capacity, maximum breathing capacity and blood pressure.

Subjects, presumedly well-conditioned to strenuous exercise, were picked with the help of the athletic department. Controls consisted of an equal number of medical students of approximately the same height and weight as the athletes.

Control readings were taken after a thirty minute rest period. Each subject was then put through a standard amount of exercise on a stationary bicycle and the ballistocardiographic and blood pressure studies being repeated immediately following the exercise.

The data obtained indicate that the ballistocardiograph may be used for the estimation of cardiac stroke volume following exercise if the post-evercise records are normal contour. It was also found that the well-trained athlete exhibited less change in heart rate, stroke volume and blood pressure than the untrained person. After exercise the athlete also demonstrated a faster return to normal of all the cardiovascular and respiratory factors studied.

\*This project was carried out by the aid of the Alpha Phi Heart Fellowship.

#### A COMPARISON OF DIGESTIBILITY COEFFICIENTS BETWEEN THE RAT AND THE PIG

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Chromic oxide is being widely used as an inert reference substance in the determination of digestion coefficients. The use of chromic oxide for this purose has advantages over the old conventional method. It eliminates the error due to the waste of feed and the loss of feces during the collection period and also eleminiates the error due to difference in moisture between the feed and feces. For determination of coefficients of digestibility of nutrients it is only necessary to know the chromic oxide ratio of the particular nutrient in the feed and feces. From these two ratios the coefficient of digestion can be easily determined. In view of these advantages the chromic oxide technique was used for comparing the digestive coefficient of a ration obtained by feeding the rat and pig the same type of ration.

It is always an advantage to use a smaller animal in place of a larger animal for experimental purposes. Before a rat can be used to measure the coefficient of digestibility of a feed which is to be used for feeding the pig, it is necessary to know whether the digestive coefficients of a feed are of the same value for the two animals. If this relationship between the rat and pig is known, the rat can be used to measure digestive coefficient of a feed for a pig. This could quickly give an index of the nutritive value of a feed from rat trials. Such information could result in an economic advantage. If the rations did not meet the nutritive requirement of the rat, it could be so modified to meet the nutritive requirements before being fed to pigs.

This study was undertaken to obtain this information with the aid of chromic oxide techniques; the results of which are presented.

Ten growing pigs and twenty growing rats were fed a basal barley ration. One-half per cent chromic oxide was added to the ration as an inert reference indicator. After the rats and pigs were fed this ration for a week, samples of feed and feces were collected, dried and ground in a Wiley mill and analyzed for protein, carbohydrates and organic matter. The protein was determined by the Kjeldahl method. The carbohydrates were determined by hydrolyzing a 500 milligram sample at room temperature with 60 per cent sulfuric acid. The sample was then diluted to 500 ml. with distilled water. Any residue not in solution was allowed to settle and one ml.

aliquot was taken and analyzed colormetrically for sugar with anthrone. Pure dextrose was used as a reference standard.

The per cent organic matter was determined indirectly by subtracting the percent ash and moisture from 100.

The per cent chromic oxide was determined by the modified method of Bolin et al.

The results of feed analyses for the four different rations (Table I.) are approximately the same. This is to be expected since the only difference in these rations is that a small amount of amino acids were added. The use of these rations should serve to indicate the precision of the methods used for the determination of the digestion coefficient. The addition of the amino acids would normally have little effect on the digestion coefficient.

In Table I the analyses are shown for the feeds and for the rat and pig feces. Because of similarity of rations there was very little variation in chemical composition for the rat or for the pigs. There is some variation between the pig and rat, which would indicate the rat digested his rations better than the pigs. This statement is also verified from the digestibility coefficient shown in Table II.

These differences between the rat and the pig are very small. This small difference in digestion coefficient may be due to other factors than digestion of the ration. For example; wood shavings were used for bedding and it is possible that a few shavings may have been consumed by the pigs. This would lower the digestibility coefficient. In conclusion, the results of this data presented should be in no way interpreted that the digestibility coefficient are the same at all times for the pig and rat. These results indicate only, there is a correlation in the digestion coefficient for the rat and pig on this particular type of ration. This correlation may vary on different types of ration. More experimental work of this nature is needed before any definite conclusions can be drawn. Such experiment can be easily carried out in conjunction with other experiments, if the chromic oxide technique is used.

TABLE I

The Per Cent Chemical Composition of Feed and Feces
Ration Number Protein Carbohydrate Organic Matter Chromic Oxide

		•	-	
				Oxide
	%	%	%	%
I	12.35	65.14	88.98	.542
II	12.40	65.41	88.95	.548
III	12.30	65.40	88.95	.522
IV	12.30	65.10	88.82	.529

		Rat Feces		
Ration Number	Protein	Carbohydrate	Organic Matter	Chromic
				Oxide
	%	%	%	%
I	17.00	25.42	82.90	2.246
II	16.45	25.89	83.16	2.246
III	17.00	26.15	83.30	2.165
IV	17.05	26.01	83.30	2.209
		Hog Feces		
Ration Number	Protein	Carbohydrate	Organic Matter	Chromic
				Oxide
	%	%	%	%
I	15.12	26.15	81.48	2.06
II	15.14	26.40	81.69	2.06
III	15.90	26.54	81.57	1.94
IV	15.34	26.06	81.63	2.09
	10.01	20.00	01.00	2.00

TABLE II
Percent Digestibility

Ration	Protein		Carbo	hydrate	Organi	Organic Matter	
	Rat	Pig	$\mathbf{Rat}$	Pig	Rat	Pig	
1	67.07	67.79	90.58	89.44	77.52	75.91	
II	68.08	67.52	90.34	89.27	77.19	75.57	
III	66.68	65.22	90.36	89.08	77.42	75.32	
IV	66.81	63.43	90.43	89.87	77.54	75.74	
Average	67.16	65.99	90.43	89.42	77.42	75.64	

### A NEW PROCEDURE FOR SCREENING NURSERY WHEAT SAMPLES FOR MILLING QUALITY

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#### ABSTRACT

The procedure described by Bruner and Harris in the 1955 Proceedings of the Academy of measuring flour volume was applied to wheat meal with the objective of evaluating the possibility of using it for screening plant breeders' samples for milling quality without actually milling wheat. Essentially the method involves the determination of the volume of whole wheat ground in a Labconco laboratory mill as commonly used for nitrogen tests.

Positive correlation coefficients greater than 0.93 were found between flour volume and wheat meal using 25, 50 and 75 grams of the meal in a series of 32 samples. For a series of 64 samples the corresponding values with the 25 and 50 gram methods were above 0.91. These high relations indicated that wheat meal volume should be as useful as flour volume for assaying milling quality. This conclusion was verified by high negative correlations between flour yield and flour and wheat meal volume (0.81 and 0.79 respectively). There was no relation between the test weight of the wheat and flour yield.

The results open up the possibility of using the relatively simple and rapid wheat meal volume test as a means of eliminating hybrids of unsatisfactory milling quality at a early stage of the breeding program.

#### ON ORIGIN OF LEONARDITE

by N. N. Kohanowski

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Leonardite is a coal-like substance often found in association with lignite in North Dakota. It was named after A. G. Leonard who headed the Department of Geology at the University of North Dakota and also was the first director of the North Dakota Geological Survey. Its presence in lignite is objectionable since leonardite lowers the heating value. On the other hand, leonardite may be utilized in water softening and as a porosity sealer in drilling muds. Experiments are going on in regard to utilization of leonardite as an additive in fertilizers and as a binder in pelletization of taconite. The price of leonardite has doubled on the market in the past three years.

This paper is based on the field and laboratory investigations which were carried on as one of the projects of the North Dakota Research Foundation during the past three years. The purpose of investigation was to find the mode of formation and occurrence of this material which may be both objectionable and useful.

Long thought to be a form of lignite oxidation, leonardite actually forms independently of lignite. It is leached by alkaline waters out of top soils of the gumbo type, transferred into subsurface and precipitated whenever the pH value of waters falls below 6.5. Contact of solutions with lignite hastens precipitation but is not absolutely necessary.

Three types of leonardite are distinguished.

**Type I** is a mixture of leonardite II with lignite. Admixed lignite is fragmented and shows but a little corrosion, which is largely due

to removal of gel material leaving the cellular structure of plant tissues intact. The mixture occurs in fragmented seam tops or along water courses. It is found wherever lignite seam is overlain with less than an eight foot thickness of water permeable sandy sediments. Greater thicknesses of such overburden and clayey overburdens have protected lignite in most cases seen. A few exceptions, like that of the Miller mine (near Sawyer) may be explained by leonardization in the past when the overburden was very much thinner.

Type II leonardite is analogous with what has been described in the earlier editions of the Dana's System of Mineralogy under the name of Native Humus Acid. It is a black colloidal material which swells in water to several times its original volume and is soluble in alkali hydroxides almost without a residue, giving dark brown, rich colored solutions. It is this property of swelling that makes leonardite utilizable in drilling muds. On dehydration, leonardite hardens, temporarily adquires a vitreous luster, then becomes dull and cracks. Hardened leonardite II is apparently identical with tucholite of North Saskatchewan except for low radioactivity or its entire lack. In laboratory conditions dissolved leonardite II may be reprecipitated as a light brown colloid, not unlike ferric hydroxide in appearance, with hydrochloric acid at pH of 4 or lower. In nature, precipitation taken place at pH values up to 6.5 and the precipitated material is dark brown to black. The reason for this inconsistency of laboratory evidence with the field observation has not been ascertained. It may possibly be due to action of lignite or other organic matter which reduces humic acid to elemental state. This view is supported by existence of "paper coal"—thin sheets of elemental carbon which are occasionally found in fractures in the top part of the Velva mine seam. On drying these sheets curl up and may be separated. On treatment with alkali hydroxides small amounts of humic acid are dissolved, the remainder being pulverulent carbon without any vegetal tissues.

The **type III** of leonardite, as found in the Government pit of Peerless mine is similar to the laboratory re-precipitated light brown substance. It is very fine grained, colloidal and is intimately intergrown with gypsum (CaSO.2H<sub>2</sub>O). Intergrowth with gypsum indicates that leonardite III was precipitated in presence of the sulfate anion. The source of calcium is thought to have been Dopplerite, a calcium humate, although the latter has not been identified positively.

Averages of chemical analyses are shown in the table 1. The organic analyses were made by Dr. G. A. Abbott and the ultimate and proximate analyses by Miss Adelain Magnusson, the fuel analyst, both of the University. The ultimate and proximate analyses are given on moisture and ash free basis.

TABLE 1. Average Analyses. (Percenta)

	Leo	nard	ites	N. D.	Penna.
	I	II	III	Lignite	Bituminous
Volatile and					
combustible	79.82	85.25	83.55	90.87	97.68
Ash	20.18	14.51	15.45	9.12	2.32
Benzene extract	0.79	0.16	Trace	Trace	0.39
Alcohol (95%)					
extract	2.79	0.87	1.75	1.80	1.72
HCl extract	11.71	14.68	16.84	1.53	16.84
Humic acids in					
organic, etc.	44.51	81.38	9.35	9.53	0.102
Volatile matter	50.75	57.25		47.50	
Fixed Carbon	49.25	42.75		52.50	
B. T. U.	10,130	9,550		11,930	
Carbon	64.57	60.91		68.07	
Hydrogen	4.03	4.07		4.75	
Sulfur	0.48	1.35		0.81	
Nitrogen	1.11	1.15		1.03	
Oxygen	29.81	32.52		25.34	

The proximate and ultimate analyses indicate that leonardites occupy an intermediate position between the ranks of peat and lignite. Although not readily realizable on basis of these analyses, leonardites differ from peats in their greater content of humic acid. Presumably this is due to a greater decomposition of vegetal matter and parallels observations in the present day swamps of northern Saskatchewan and in highlands of Colorado.

The humic acid content, as determined by Dr. Abbott, decreases rapidly with the rise in rank. Qualitative analyses by the writer have shown a total absence of humic acid in authracite (Donbass, U.S.S.R.) and in graphitic anthracite (prov. Fooh-Ken, S. China). This suggests that humic acids are reduced to elemental carbon. This conclusion in turn is borne out by observation at the Kincaid mine (Burke county), where colloidal coal, devoid of vegetal tissues, flowed around faulted sections of "slate" (suspension of clay in resins). The "paper coal" of the Velva mine—paper thin laminae lying loose in cleats of ordinary lignite, seems to have had the same origin.

The relationship of humic acid in top soils and leonardite is clearly visible in the Perkins pit of the Peerless mine (Gascoyne, Bowman county). There, due to a recent regional tilt, the leonardite seam comes to the surface at  $5\frac{1}{2}$ ° angle. At the junction with the gumbo topsoil the transition of the soil into leonardite is so gradual that the two cannot be delimited.

Much remains to be worked in regard to geochemistry of leonardite and its conversion into lignite. What has been done so far amounts barely to a framework for future investigations.

The idea that lignite reverts back to leonardite in some retrograde process should not be entirely discounted, although evidence against it is accumulating. Thin stringers of lignite which do not carry water but weather "in dry", do not contain humic acid. Instead, volatiles and solubles become lost and such lignite adquires a submetallic luster and approaches in composition that of schungite (an impure graphite from Siberia). Weathering of water carrying lignites in arid conditions does leave pulverulent humic acid on outcrops. This, however, is due to evaporation of ground waters and not to oxidation of lignite itself.

Chemical processes operating within leonardite seams are both numerous and complex. Three major groups of breakdown products are indicated. Humic acid and its calcium salt, dopplerite, may be totally reduced to elemental carbon, this contributing to enrichment of coal. Secondly, both humic acid and dopplerite existing in leonardites I and II may be converted to leonardite III by sulfatic waters. At the time this paper was sent to print it became apparent that on digestion of leonardite with sulfatic waters there forms not only gypsum but an insoluble ferrous humate as well. Thirdly, leaching of leonardite II results in formation of organic minerals such as acetates of ferrous iron, aluminum and calcium.

Thickness and distribution of leonardite seams in the State are in harmony with climatic conditions visualized during the Pleistocene glaciations. In the northwestern part of the State individual leonardite seams and laminae are thin, from a fraction of an inch to perhaps a little over a foot. Thicknesses increase southward and reach a maximum of six to eight feet in Bowman county. The shape of leonardite seams appears to be lenticular with abrupt, pinched out or "slumped" extremities.

With the exception of leonardite laminae of the Kincaid mine, leonardite lies mostly in unconsolidated Pleistocene sediments. At the Peerless mine partly carbonized tree stumps in upright positions and non-carbonized grass roots were found within the upper part of the leonardite seam, the grass roots resembling those of quack-grass. Bison bones were found in association with lumps of leonardite II in lacustrian sands which cover the lignite seam (of possibly Hell Creek age) in the 1956 pit of the Dakota Briquette and Tar Products Co. near Dickinson.

The cumulative picture of vegetation during glaciations of Pleistocene resolves in the following manner.

In the northwestern corner of the State small isolated patches of vegetation existed sporadically at the very boundary of the ice sheet. Southeast of Minot in the Velva-Quality mines area, vegetation was fairly moderate in intensity in vicinity of lakes. The area southwest of the Altamont moraine, as represented by deposits of Custer, Dakota Star and Zapp mines was a grassland. Lakes surrounded by grassland existed in the vicinity of Dickinson. On the south, in the area of Bowman to Haynes there developed swamplands disected by meandering streams.

### FORAGE PRODUCTION ON GRAZED RANGES IN RELATION TO COMPOSITION OF THE VEGETATION<sup>1</sup>

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#### ABSTRACT

Production of forage on eight grazed range areas in the mixed grass vegetation type of western North Dakota has been determined for the 7-year period, 1950-1956. Clippings from movable steel cages were used to determine total yield. Species composition of the yields was estimated. Composition of the vegetation on each of the range areas has been determined by means of point analysis. Height growth of the principal species in each area has been determined for the period 1953-1956.

All range areas sampled were grazed in the summer, with the principal period of grazing extending from May to October inclusive. The sites of the sampling areas are somewhat different in each case, but in general all would qualify as "ordinary upland", in the Soil Conservation Service's Technicians Guide. Vegetation is essentially similar on all areas, with compositional differences measurable principally in differences in percentages of the same species.

Results of the study show that the lowest producing areas had an average yield of 818 pounds of dry material per acre for the 7-year period, while the highest producing areas averaged 1467 pounds per acre for the period. On the highest producing areas the midgrasses made up about 70 percent of the yield and on the lower producing areas about 53 percent. Short grasses produced 25 percent of the yield on the high-yielding ranges and 37 percent on the low-yielding ranges.

Basal area analyses of the cover of the sampling areas showed that the high-yielding areas averaged 17.6 percent mid-grasses, while the low-yielding areas had 9.1 percent of mid-grasses in the cover. Yield was apparently closely and directly related to percentage of mid-grasses in the cover. On the high-yielding areas short grasses made up 65 percent of the cover and on the low-yielding areas 83

percent. Yield was apparently closely but inversely related to the percentage of short grasses in the cover.

Plant vigor was lower on the low-producing areas than on the high-producing areas. This is shown by the average height of the leaf growth of the three principal grasses on the sampling areas. The three principal grasses, western wheatgrass, needle-and-thread, and blue grama grass, averaged about 25 percent shorter on the low-yielding than on the high-yielding areas. As would be expected, the relation between leaf-height of the principal grasses and yield is close and direct.

The spread in differences in yield on the grazed range areas is sufficient to account for a difference rating of nearly two full condition classes. While it is difficult to assess accurately the significance of composistional differences and vigor differences, it would appear that about two-thirds of the yield difference between the low and the high-yielding areas is the result of differences in composition of the vegetation. About one-third of the yield difference is apparently due to differences in plan vigor between the areas.

1. Published with the approval of the Director, North Dakota Agricultural Experiment Station.

# A BRIEF ANNOTATED LIST OF PUBLICATIONS PERTAINING TO THE BIRDS OF NORTH DAKOTA, WITH SPECIAL REFERENCE TO THE RED RIVER VALLEY

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The purpose of this paper is to furnish a list of the books and published articles the compiler has found most helpful during his observations of birds in this region. This is not a complete bibliography, and will not serve the purposes of scientific ornithology. But the list should be useful to teachers and students, or to newcomers in the region who share the compiler's interest in the general study of birds.

#### I. GENERAL PUBLICATIONS

For bird study in any region, indispensable aids are a field guide, a good set of color pictures (plates), and one of the larger books on birds. The following are recommended:

PETERSON, R. T. A Field Guide to the Birds. 2nd. ed. Houghton Mifflin Co. Boston. 1947. The classic guide for field identification of species. This is the so-called "eastern" guide. The companion western guide is scarcely required in the Valley, although it may be needed in western North Dakota.

TAVERNER, P. A. Birds of Canada. David McKay Co. Philadelphia. 1940. This I regard as the most satisfactory general "bird book" for our region, because of the author's close acquaintance with many species as they live in the nearby and ecologically similar prairie provinces of Canada. Good color plates by Allan Brooks and F. C. Hennessey.

FUERTES, L. A. Color plates in Birds of America. T. G. Pearson, editor. Garden City Publishing Co. Garden City, N. Y. 1936. The most economical set of good plates currently available. (The well-known Audubon color plates are not as useful for bird study as are paintings by such later artists as Fuertes or Brooks.) The text material in this book is unfortunately rather unrewarding and outdated.

The books listed above are at present in print.

#### II. REGIONAL LISTS AND OTHER REGIONAL STUDIES

WOOD, NORMAN. A Preliminary Survey of the Bird Life of North Dakota. University of Michigan Museum of Zoology. Misc. Publications No. 10. Ann Arbor. 1923. The author visited North Dakota, including the Valley, in the summers of 1920 and 1921. This list is based on these visits, plus extensive bibliographical work (bibliography is given). This is the only authoritative published bird list for the state. It is prepared by a recognized ornithologist, but not by one with an intimate year-by-year acquaintance with the bird life of the region.

MONSON, GALE W. The birds of Berlin and Harwood townships, Cass, County, North Dakota. Wilson Bulletin, Vol. XLVI (1934), pp. 37-58. Describes the occurrence, migration dates, and relative abundance of species in the part of the Valley mentioned. Based on intimate observations and daily records extending over a period of years. It is, as a result, the most useful aid for the student of birds in the Valley. (For water birds, the records are incomplete, because there was no permanent wetland in the area.)

STEVENS, O. A. A migration list from Fargo, North Dakota, 1910-1949. Flicker, Vol. 22 (1950), pp. 90-104. A tabulation of dates for many species observed or trapped by the author in Fargo.

WILLIAMS, H. V. Birds of the Red River Valley of northeastern North Dakota. Wilson Bulletin, Vol. XXXVIII (1926), pp. 17-33 and 91-110. Lists records and remembrances of W. H. Williams and H. V. Williams, father and son, bird students and taxidermists of Grafton, N. D.

The Wood list may still be available from the University of Michigan. The Monson and Williams publications are no longer available as separate reprints, but single copies of the Wilson Bulletin may be available at low cost from the Museum of Zoology, University of Michigan. The publications listed above are in the libraries

of the North Dakota State College at Fargo and (except the Williams list) of the University of North Dakota at Grand Forks.

Of other lists for the state, E. T. Judd, List of North Dakota Birds Found in the Big Coulee, Turtle Mountains and Devils Lake Region, privately printed, Cando, N. D., 1917, pertains to a region close enough to the Valley that it is of interest here. It is out of print. The list by A. Larson, Wilson Bulletin, Vol. XL (1928), pp. 39-48 and 100-110, for McKenzie County; and the pamphlet by O. A. Stevens, Birds of the Theodore Roosevelt National Memorial Park Area; describe bird life on the western edge of the state.

#### III. EARLY PUBLICATIONS

For those interested in early records of bird life in North Dakota, a few references are given in this section. The earliest scientific exploratory expeditions into the Dakota Territory were those of Lewis and Clark (1804-6), Maximilian (1833), and Audubon (1843). The journals of these expeditions are not confined to birds. All these travelers went up the Missouri River. The earliest overland parties to include naturalists were the Northern Pacific expedition across the western part of the state in 1873, which was accompanied by J. A. Allen, and the Hayden international boundary survey party of 1873. Elliott Coues accompanied the latter party, and his records from the trip are about the earliest ornithological records from the Valley. However, only about a month of the time was spent in the Valley (at Pembina). (The term "Red River region" is used in early publications to cover a more extensive geographical area than is today implied by "Red River Valley").

LEWIS, MERIWETHER. Lewis and Clark in North Dakota, edited by Russell Reid. State Historical Soc. of North Dakota (reprinted from North Dakota History). Bismarck, N. D. 1947-8.

MAXIMILIAN. Travels in the Interior of North America, by Maximilian, Prince of Wied. Vols. XXII-XXIV of Early Western Travels, edited by R. G. Thwaites. A. H. Clark Co. Cleveland. 1905-6. (Out of print.)

AUDUBON, J. J. Audubon and His Journalis, edited by Maria R. Audubon and E. Coues. 2 vols. C. Scribner's Sons. New York. 1897. (Out of print.) HARRIS, EDWARD. Up the Missouri with Audubon. The Journal of Edward Harris, edited by J. F. McDermott. Univ. of Oklahoma Press. Norman, Oklahoma. 1951.

ALLEN, JOEL A. Notes on the natural history of portions of Montana and Dakota. Proceedings Boston Society of Natural History, Vol. 17 (1874), pp. 14-38.

COUES, ELLIOTT. Field notes on birds observed in Dakota and Montana along the forty-ninth parallel during the seasons of 1873 and 1874. Dept. of Interior U. S. Geol. and Geog. Survey of the Territories Bulletin, Vol. 4 (1878), pp. 545-661.

#### IV. OTHER PUBLICATIONS

JOHNSGARD, P. A. Waterfowl of North Dakota. N. D. Institute for Regional Studies, Fargo, N. D. 1953. Summarizes occurrence, relative frequency, and other information on waterfowl, and gives drawings and marks for identification. The Monson and Stevens papers listed in part II above are incomplete for water birds, because of lack of water in the local areas covered. The Valley is not slough country, but there are some wetland areas, and the Valley bird observer may have use for this pamphlet. It is to be revised and reissued.

No competent ornithological bibliography for the state has been published (except the bibliography in the 1923 Norman Wood list). A typewritten copy of a bibliography by Russell Reid, including references through 1928, is in the State College library at Fargo. (A compilation by T. C. Stephens, issued in mimeographed form in 1956 as Occasional Papers, No. 2, of the Nebraska Orithologists' Union, gives an incomplete list of references through 1947.)

Dr. J. Frank Cassel informs me that an extensive list of dates and places of occurrence of birds in North Dakota, by W. C. Bell, is now being copied in the Zoology Department at N.D.A.C. When this work is completed, typewritten copies will be deposited in the State College and University libraries.

The popular publication by Wilford Miller, Wildlife in North Dakota, Bismarck Tribune Co., Bismarck, N. D. 1955, contains an unannotated list of birds that may occur in the state, with subspecies not distinguished from species except by the Latin trinomials. However, it contains some general information on game species, and maps showing location of wildlife refuges, which may be of interest.

The publications cited thus far are essentially all species reccords and lists of occurrences. Dr. Cassel's group at Fargo is at present conducting some studies along lines of more current interest to modern ornithology, and some of their results are beginning to be published—for instance J. F. Cassel, Winter bird populations in the deciduous forest along the Red River in North Dakota, abstracted in Proceedings of the North Dakota Academy of Science, Vol. X (1956), p. 12; and C. Weiser and D. J. Hlavini, Comparison of breeding-bird populations of three deciduous shelterbelts, Audubon Field Notes, Vol. 10 (1956), pp. 416-19. In the latter study, the shelterbelts were located in the Valley north of Fargo.

As basic aids for bird study in the Red River Valley, I would recommend, besides some books such as those listed in part I, the Monson and Wood papers cited in part II. Next, probably, would come the Stevens and Williams lists (see part II), and the Johnsgard pamphlet (see part IV, above).

# DISTRIBUTION OF PHYTOCLIMATE INDICATORS ON CONTRASTING SLOPES OF THE FOREST RIVER, NORTH DAKOTA

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The vegetation on contrasting slopes in the Forest River area, Grand Forks Co., was investigated during the summers of 1954-56. The percentage vegetational cover was estimated for each species along transects by the line interception method, while the phytoclimate indicator class was determined in the field according to Raunkaier's system. At regular intervals along the transect soil samples were obtained for the determination of water retaining capacity and pH. Soil temperature readings were obtained at the same sites as were those for relative humidity and light.

The results indicate a higher percentage of phanerophytes and lower percentage of hemicryptophytes on the north-facing slopes. Associated with these results are higher water retaining capacity of the soil, variation in pH from that of the adjoining prairie, less available light, and higher relative humidity. Cryptophytes with their over-wintering buds in the soil were almost equally segregated on both slopes, while annuals (Therophytes) were confined to the disturbed areas along the river.

### AN OCCURRENCE OF BURIED CONIFEROUS WOOD IN THE ALTAMONT MORAINE IN NORTH DAKOTA'

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Samples of wood exposed in an excavation in the Altamont moraine south of Tappen, North Dakota have been sectioned and tentatively identified as wood of white spruce. Female cone material from the same site has been definitely identified as white spruce. Both types of material are in a good state of preservation; the depth at which they occur, approximately 15-18 feet below the surface, precludes the possibility of recent origin.

Detailed stratigraphy of the site has not yet been worked out, but the evidence to date suggests that the material grew in this site and was subsequently buried in glacial debris. The surface glacial

deposits of this part of the Altamont moraine are probably of Mankato substage, underlain by Cary drift.

The discovery of this coniferous wood implies a climate cooler and moister than is currently experienced in that area. At the present time the closest native stands of white spruce are in the Black Hills about 200 miles southwest; in the Spruce Hills area of southern Manitoba, about 200 miles to the north; and along the edge of the coniferous forest in Minnesota about 200 miles to the east.

Radiocarbon dating of the wood indicates an age of approximately 11,480 years, correlating with the Two Creeks interstadial which occurred between the Cary and Mankato substages of the Wisconsin glaciation.

#### INTRODUCTION

In the fall of 1955 a site containing buried wood was uncovered by a power-shovel operator while attempting to open up a possible water source in a pasture on the Wolt farm about 10 miles south of Tappen, North Dakota. The exact location is in a draw about 200 feet southwest of the center of section 25, T. 138 N., R. 71 W., in Kidder County. This draw carries intermittent surface drainage to the northeast from the rolling hills of a segment of the Altamont moraine. In earlier work with a hand augur and a tractor-mounted scraper, Mr. Wolt had exposed wood fragments and other vegetative debris in the surface layers. When the power shovel was being used to deepen the excavation, a large stump was contacted at about 15 to 18 feet below the surface. The stump was approximately 10 inches in diameter and in an upright position. The base was embedded so firmly that it interfered with the operation of the power shovel, and could not be removed. Large pieces of the material were broken away and brought to the surface with the wet sandy colluvium being removed from the excavation.

Because no increased flow of water was obtained as a result of the excavation, and the fine sand composition of the valley floor caused the walls of the excavation to slump in as fast as the wet sludge was removed, no further attempt was made to deepen the excavation.

Some time after the excavation had been abandoned, it was observed that through drying and slumping of the sides of the pile of material removed from the excavation, coniferous cones were being exposed. Until this time the presence of the stump had not been considered to be of any significance, but the discovery of the cones in an area far removed from any native stands of coniferous trees attracted the attention of the land owner.

#### PROCEDURE

A preliminary inspection of the site in late November of 1955 indicated that it was highly improbable that the wood and cones were of recent origin. Samples of both of these materials were ob-

tained at that time, and during the summer of 1956 hand and power drilling equipment was utilized to obtain more information on the stratigraphy of the site.

Specimens of the wood of varying size, some from the main trunk and others from smaller roots and branches were soaked in a water-glycerine mixture until soft enough to allow sectioning. Sections of about 30 microns thickness were cut with a hand microtome and made into permanent slides for identification. The wood sections showed remarkably good preservation of cellular detail, and have been identified as spruce. As there is little structural difference between white and black spruce, identification as to species has not been definitely established. However, its association with cones definitely identified as white spruce, leaves little doubt that the wood is of the same species. The size of the growth rings indicate that environmental conditions favored rapid growth.

Samples of the sand and clay material from the level where the stump was located were examined for pollen content. These samples were obtained from the sandy debris adhering to the wood samples, and by using a soil augur to remove clay samples at a depth of approximately eighteen feet below the surface of the undisturbed valley floor in the vicinity of the excavation. Unidentified pollen and spores in low concentration were observed, but as yet no coniferous pollen has been found.

Hand drilling to a depth of 18.5 feet, and machine drilling to 63 feet, revealed the following profile:

From	To	Nature of Material
0′	1'	Turfy material in fine black matrix.
1'	9′	Sand, black to gray, uniform, well-rounded; variable water content.
9′	10′	Wood fragments, unidentified plant remains suggesting aquatic vegetation in a matrix of dark gray clay silt.
10'	15′	Gray sand as above, with gastropod shells and wood fragments from 12' - 15'.
15′	18′	Gray sand, fine, well sorted, rounded, becoming more compacted and with higher clay content approaching 18'.
18′	34′ ;	Clay, light gray; top layer very sticky when wet, drying to a hard brick-like material (lowest level of hand-drilling 18.5 feet)
34'	40'	Till, dark gray.
40'	47'	Gravel, pea size with gray sand lenses.
47'	54'	Gravel with cobbles, water.
54'	57'	Clay till.

Fox Hills sandstone.

63'

57'

Radiocarbon dating of a wood sample from this site submitted to the U. S. Geological Survey Geochronometric Laboratory by R. W. Lemke of the U. S. Geological Survey indicates an age of 11,480 years  $\pm$  300 years. The significance of this dating is discussed in the following section.

#### DISCUSSION OF RESULTS

The approximate maximum advance of the Wisconsin glaciation is marked in the Dakotas and adjacent Canada by the Altamont terminal moraine complex, extending in North Dakota from the northwest corner of the state in a generally southeast direction and passing into South Dakota about 150 miles west of the eastern boundary of the state in McIntosh County. This massive glacial feature forms a belt of hills varying in width from 10 to 20 miles, essentially uninterrupted through its entire length.

It has long been recognized that the "Altamont" moraine through its entire extent is not necessarily equivalent to the Altamont moraine at its type locality in eastern South Dakota. Various workers. including Townsend (1), Townsend and Jenke (2), Flint (3), Leverett (4), and Chamberlin (5), have discussed the complexity of its origin, and even suggested alternate nomenclature for certain segments. It is beyond the scope of this paper to review all reports on this moraine, except to point out its anomalous structure in some segments as compared to the usual concept of a terminal moraine. In numerous places a bedrock high is present forming the core of the moraine. In the vicinity of the buried wood site the veneer of glacial till is variable, or even absent in some localities which show exposures of Fox Hills sandstone. From the limited data available, the pre-glacial topography of the area suggests a badlands aspect, with some very deep valleys, at present filled with glacial debris. Successive advances of the Wisconsin ice have greatly modified the pre-glacial topography, but it is not difficult to visualize earlier interstadials of more rugged topography than at present, presenting a great diversity of habitats and striking microclimatic differences. This would be expecially true during the periods immediately following ice recession.

The presence of spruce in this area, not necessarily as continuous forest cover but more probably in scattered groupings in the more favorable habitats, indicates a climate cooler and moister than that experienced at the present time. The closest native stands of spruce in the current distribution pattern are in the Black Hills at out 200 miles to the southwest; along the edge of the coniferous forest in Minnesota about 200 miles to the east; and in the Spruce Hills area of the Assiniboine River valley, again about 200 miles to the north in Manitoba. It is interesting that in this latter outlier of the northern coniferous forest the trees are not present as a continuous forest cover, but rather in an open woodland of separate stands of spruce.

The radiocarbon dating of this material at  $11,480\pm300$  years correlates with the dating of plant material from the Two Creeks forest bed site in Wisconsin. The average of several determinations from this site is  $11,404\pm350$  years. The Two Creeks interstadial is accepted generally as the interval between the Cary and Mankato substages of the Wisconsin glaciation. The correlation of these dates would suggest that the spruce trees of the Kidder county site described here also grew during the Two Creeks interstadial, and were later buried by the accumulation of till and outwash material from the Mankato advance at its maximum. In more recent times alluvium and colluvium carried in from the adjacent hills have added to the overlying layers.

This find is of interest because relatively few datings are available from our region. A discovery discovery discussed by Flint & Deevey (6) from a sewage disposal excavation in Lake Agassiz deposits on the banks of the Red River at Moorhead revealed a great variety of plant materials, including larch and white spruce. The first dating on this wood was  $11,283 \pm 700$  years, but a more recent determination of 9970 years is more in line with what might be expected, since the material was found in Lake Agassiz sediments and some time lapse from the Mankato maximum would be necessary to allow the accumulation of sediments on the lake bottom below the sample. It is suggested that this material was carried by the Buffalo River from beaches and moraines some 20 miles to the east.

Other finds, either undated or pre-dating the Two Creeks interstadial have been described in Minnesota, South Dakota and Manitoba, but like the Moorhead site none of the materials involve trees in situ, as is suggested by the Kidder county material.

### SUMMARY

Samples of a coniferous tree stump and associated cones uncovered in an excavation in the Altamont moraine in Kidder County, North Dakota, have been identified as white spruce. Radiocarbon dating of a sample of the wood, believed to be **in situ**, yield an age value of approximately 11,480 years, correlating closely with the Two Creeks interstadial. This material was evidently buried by sandy outwash that may date the maximum advance of the Mankato ice, and its presence here suggests a climate cooler and moister than is observed at present.

### ACKNOWLEDGMENTS

The writer wishes to acknowledge the assistance of W. M. Laird and G. L. Bell of the N. D. Geological Survey, and R. W. Lemke of the U. S. Geological Survey for obtaining the drill log at the site, and the radiocarbon dating of the wood, and the further assistance of G. L. Bell in the interpretation of the preglacial topography. The assistance and hospitality of the landowner, Mr. Walter Wolt, is also much appreciated.

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- 1. Funds for field work on this project were provided by the Dept. of Botany and the Institute for Regional Studies, N.D.A.C.

# HELIUM II: A LIMITATION ON VARIATIONAL PROCEDURES

by Kenneth Hartt

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Correctly describing the peculiar properties of helium II poses many challenging problems in theoretical physics. It is generally felt that the equilibrium properties, such as the lambda transition, can be understood once we know how a quantum fluid can absorb energy. We cannot, unfortunately, proceed in a straightforward way in answering this question. That would entail finding the infinitely many solutions to a differential equation in some  $10^{2i}$  variables. Therefore, one must rely upon physical intuition to form some advance concept of the solution. The purpose of this note is to show why one mathematical tool, the variational method, cannot be used as a valid criterion for comparing physical "guesses" about energy absorption. It appears worthwhile to present this limitation because of a recent plausibility argument which contained such a method.

States of a physical system may be represented by vectors in an infinite dimensional space which is a generalization of the space in which we may give, say, the velocity of a rocket as a three-dimensional vector. This generalization is called hilbert space, and is technically characterized as being a linear, metric, countable, separable

and complete vector space. We refer to von Neumann's book, Mathematical Foundations of Quantum Mechanics, Ch. II, for the postulates and definitions. For our purpose it suffices to mention only that distance between vectors  $\mathbf{f}$  and  $\mathbf{g}$  is defined such that a quantity  $(\mathbf{f},\mathbf{g})$  (read: inner product of  $\mathbf{f}$  and  $\mathbf{g}$ ) is inversely proportional to their distance of separation. By a variational procedure we mean the determination of the values of a set of parameters  $[a_1,a_2,\ldots,a_n]$  for which a function  $\mathbf{f}(a_1,\ldots,a_n)$  is minimized or maximized:

$$\frac{\partial f}{\partial a_1} = 0, \qquad i = 1, 2, \dots n. \tag{1}$$

That function we will take to be  $(\psi(a), H \psi(a))$ , which we will abbreviate as (H). H is the hamiltonian, the differential operator of the Schroedinger equation:

$$H\phi_i = \lambda_i \phi_i$$
,  $i = 0, 1, 2, ...$  (2)

 $\psi(a)$  is a trial solution for which the best value of the a's is sought. For a given  $\psi$ , (H) has the physical interpretation as the average energy in that state.  $\phi_{\circ}$  is the solution corresponding to the lowest energy state.

The usual use of a variational procedure is to determine an upper bound to the lowest energy value,  $\lambda_o$ . This is entirely justified by the fact that (H)  $\geq \lambda_o$ , always; a wide variety of calculations has been made in this manner. But one can easily show that in the framework of hilbert space such a procedure will not necessarily bring the trial vector any nearer to the true ground state vector. This is the essential import of the following theorem.

**THEOREM:** There exist linear manifolds in hilbert space in which the vector corresponding to  $(H) = \min$ , does not also have the property that  $(\phi_0, \psi_0) = \max$ . We shall just outline the proof.<sup>3</sup> Take the form of the normalized trial function to be

$$\psi = \sum_{p=1}^{n} G_{p} a_{p}$$
 (3)

where the  $G_p$  are normalized functions in configuration space and the  $a_p$  are real. Also,

$$\sum_{p=1}^{n} a^{2}_{p} = 1.$$

The  $G_p$  can be represented by their complex expansion coefficients  $[c^p_i, i=0, 1, 2, \ldots]$  with respect to the solutions  $\phi_i$  of the Schroedinger equation. That  $(H) = \min$  is expressible in terms of a set of equalities of the form

$$\frac{a_{p}}{a_{n}} = f(|c^{p_{1}}|, \lambda_{1}), \qquad p = 1, 2, \dots n-1$$
 (4)

If the signs of the ap are properly chosen, the extremum will neces-

sarily be a minimum. A similar set of equations results from the condition that  $|(\phi, \psi^{(a)})|^2$  be an extremum:

$$\frac{a_p}{a_n} = g(a_1, a_2 \dots a_n, R_0^e, I_0^e), \qquad (5)$$

$$p = 1, 2, \dots n - 1$$

where R<sup>p</sup> and I<sup>p</sup> stand for the real and imaginary portions of c<sup>p</sup>.

The argument is as follows. The solution of equations (5) depends upon the ground state expansion coefficients only, while the solution of (4) depends upon higher expansion coefficients. Therefore it is always possible to find a linear manifold in which the two solutions are not identical. An obvious corollary is that if two functions  $\chi$  and  $\phi$  have been varied in *different* manifolds to minimize (H), the values of (H) do not furnish a valid criterion for comparison.

The theorem in this note obviously does not completely vitiate the arguments of Feynman and Cohen. It simply points out a limitation in a certain kind of reasoning. Namely, a physical argument as to how to alter a trial function may produce a lower expectation value of energy, but that fact alone cannot be taken as a reason for expecting other properties of a system to be better described. One might therefore well feel that the optimistic hope expressed by the above authors' cannot be justified until other properties of helium have been successfully calculated.

- 1. Kenneth Hartt, "Liquid Helium and the Atom," North Dakota Quarterly 25, no. 1, p. 14, (1957).
- R. P. Feynman and Michael Cohen, Physical Review 102, p. 1189, (1956).
- 3. Details of this proof will be furnished upon request.
- 4. op. cit. p. 1201.

# STUDIES ON THE PREPARATION OF JERTAIN HIGHLY UNSYMMETRICAL ORGANOLEAD COMPOUNDS

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#### ABSTRACT

Triphenyllead-lithium, which can be prepared conveniently in diethyl ether by reaction of phenyllithium with PbCl<sub>2</sub> (Gilman, Summers, and Leeper, J. Org. Chem. 17, 630, 1952), can be used to synthesize organolead compounds not otherwise obtainable. Among these is triphenyl-gamma-diethylaminopropyllead, which is readily cleaved by cold dilute hydrochloric acid to diphenyl-gamma diethylaminopropyllead chloride hydrochloride, (C<sub>0</sub>H<sub>3</sub>)<sub>2</sub>Pb(Cl)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH-

 $(C_2H_5)_2$  Cl<sup>-</sup> (Gilman and Summers, J. Am. Chem. Soc **74**, 5924, 1952).

We are now investigating the possibility of extending such procedures to produce very unsymmetrically substituted organolead compounds. Such reactions seem perhaps the best approach yet indicated to the preparation of a substance which would exhibit optical activity due to an asymmetric arrangement of groups around a lead atom. In addition, the presence of the tertiary amino group might make resolution possible. By reaction of benzylmagnesium chloride with the above hydrochloride we have obtained benzyldiphenyl-gamma-diethylaminopropyllead (an oil that forms a crystalline methyl iodide derivative, which analyzes as expected). This compound on treatment with aqueous hydrochloric acid did not cleave as desired, but aqueous hydrobromic acid gave a crude product which may be the desired benzylphenyl-gamma-diethylaminopropyllead bromide hybromide. Attempts at purification have thus far given results which indicate extensive cleavage of phenyl, caused perhaps by HBr produced by decomposition of the ammonium salt group. The studies are continuing. Since an organolead compound with four different groups attached to lead is only one step away at present, the prospects of a successful outcome seem fairly good.

# QUANTITATIVE STUDIES ON THE BIOLOGICAL OXIDATION OF FERROUS IRON

# L. Dale King\*

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#### ABSTRACT

This work represents a study of the ferrous iron-oxidizing bacteria from acid mine water. The purpose of the study was to isolate and describe these bacteria and their oxidizing ability.

Controlled experiments indicated that the iron-oxidizing bacterium was an autotroph.

Iron-oxidizing bacteria, when streaked on iron agar or silica gel medium, gave rise to colonies that were dark brown, small, hard, and crusty with precipitated ferric iron.

The iron-oxidizing microorganisms are short gram negative rods, 0.5 by 1.0 microns, and nearly oval in shape. They occur characteristically in pairs although single rods may be observed. The bacteria are motile.

With a known amount of bacteria, measured on the basis of cell nitrogen, and a known amount of ferrous iron, it was possible to calculate the rate at which the bacteria utilized the ferrous iron. The decreasing amounts of ferrous iron were titrated with potassium

permanganate until the iron in the percolator medium no longer existed in the ferrous form. The cells oxidized large amounts of ferrous iron in a very short period of time.

Manometric studies were performed on the utilization of various substrates by the iron-oxidizing bacteria. When a very small amount of mercuric chloride was added to cells actively oxidizing ferrous iron, the amount of oxygen uptake quickly diminished and then ceased. When the results of the rate of utilization of ferrous iron were plotted on a graph, the characteristic substrate utilization by the bacteria was in evidence. Of the many different ferrous iron substrates used only ferrous sulfate and ferrous chloride were found to be utilized. Their rate of oxidation was comparable.

The results from this study were obtained under laboratory conditions and utilizing, for the most part, synthetic media.

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# TRYPTOSE PHOSPHATE BROTH, YEAST EXTRACT, AND ANIMAL SERA AS SUPPLEMENTARY FACTORS IN THE GROWTH OF HELA CELLS

Robert G. Fischer, George E. Kenny, and Barbara Nelson

### ABSTRACT

A strain of malignant human epithelical cells (strain HeLa) has been cultured on glass surfaces with fluid growth media containing varied amounts of tryptose phosphate broth, medium 199, and yeast extract together with animal sera and the resulting growth of the cell cultures compared with that obtained with fluid media containing human sera. Aliquots of 50,000 cells per ml. were added to culture tubes and fluid growth media consisting of human serum 10%, - M199, 90% varying to human serum 50%, - M199, 50% added to groups of cell cultures. Ten percent human serum with 90% M199 did not support cell growth for more than a few days. Media containing varying amounts of human serum, yeast extract, tryptose phosphate broth, and M199 indicated 10% human serum was adequate for cell growth if tryptose phosphate broth and yeast extract were included in the media.

The growth of poliomyelitis viruses in HeLa cells cultured in human serum necessitates repeated rinsing of the cells to avoid antipoliomyelitis antibody which might be present. Tubes of HeLa cells were cultured in media containing yeast extract, tryptose phosphate broth, M199 and 10 percent amounts of sheep serum, horse serum and chicken serum. The use of such animal sera eliminates the need for repeated rinsing of cell cultures.

# NORTH DAKOTA'S WHEAT ACRES BY ECONOMIC AREAS

# by H. L. Walster

NDAC—Institute for Reigonal Research

In spite of the fact that Federal census figures are usually considereded as dry as dust, they nevertheless present the best and most complete picture we have of the agricultural economy of the State. In 1954 I read a paper before the annual convention of the Soil Science Society of America on the theme "The Evolution of Agriculture in North Dakota." In introducing my subject today I want to refer briefly to some of the statewide calculations I made—calculations based upon wheat yields as reported annually by the Division of Crops and Livestock Estimates of the U.S.D.A.

Consider first "all wheat" which includes in addition to hard spring wheat, durum wheat, and an insignificant acreage of winter wheat. Here follows a table showing average yields by periods:

TABLE I. Wheat Yields by Periods

			Percent of Nation's Pro		
	North Dakota	<b>United States</b>	duction in North Dakota		
1895-1899	14.1	13.6	7.32		
1900-1909	11.5	14.4	8.76		
1910-1919	10.0	14.2	10.08		
1920-1929	11.2	14.0	13.31		
1930-1939	8.5	13.4	8.47		
1940-1949	15.2	17.0	13.29		
1950-1954	11.5	17.2	10.04		
1895-1954	11.5	14.8	10.75		

The last column above refers to bushels of all wheat. In the 60 years of record North Dakota produced more than 16% of the nation's wheat in 6 years.

12-16% of nation's wheat in 15 years 8-12% of nation's wheat in 24 years Less than 8% of nation's wheat in 15 years

My major concern today is to portray an answer to the question "Where in North Dakota was this wheat produced?" To get the answer I turn to the several decennial and quinquennial censuses of agriculture.

The census of 1950 grouped the counties of North Dakota into seven separate economic areas. An economic area was defined as a group of counties having "similar agricultural, demographic, climatic, physiographic, and cultural characteristics". You are referred to a "special Report of the 1950 Census" entitled "State Economic Areas: A Description of the Procedure Used in Making a Functional Grouping of the Counties in the United States."

The purpose of the paper is to present the acreages of "all wheat" and of durum wheat reported for each census as summations for each economic area.

The seven economic areas are assigned no descriptive name—you may make your own characterization with respect to wheat They are as follows:

Area 1 Area 3a Area 3b McKenzie Renville Wells Golden Valley Bottineau Eddy Billings Rolette Foster Dunn Towner Griggs Stark Cavalier Steele Mercer Ward Stutsman Oliver McHenry Barnes Pierce Morton LaMoure Slope Benson Area 3c Hettinger Ramsev Dickey Nelson Bowman Sargent Adams Area 2b Ransom Grant Richland Sheridan Sioux Burleigh Area 4 Area 2a Kidder Pembina Divide Emmons Walsh Burke Grand Forks Logan Williams McIntosh Traill Mountrail Cass McLean

Table II presents the distribution of wheat by percentage of total wheat acres in each economic area each census year. Brief comments based on Table II follow:

- Areas 1 and 2a: The Missouri Slope counties accounted for less than 10% of the State's wheat acres until after 1909.
- Area 3a: This area established itself as the great wheat area very early—by 1909 accounting for nearly one-third of the total wheat acres.
- Area 3b: Like area 3a, it, too, established itself very early, accounting for a high proportion ofthe wheat acres, but beginning with 1924 showing a rather well defined decline in the proportion of wheat acres harvested.
- Area 2b: These west central counties accounted for less than 10% of the state's wheat acres in 9 out of the 11 census years.
- Area 3c: These four counties raised a fairly high proportion of the state's wheat in 1889 and 1899, but because of greater diversification, particularly in the production of corn,

have since declined to the lowest proportion of wheat in the state.

Area 4: The five Red River Valley counties north of Richland County accounted for practically half of the State's wheat acres in 1889 and 1899, but have accounted for a steadily decreasing proportion of the wheat acres ever since except for the abnormal percentage rise in 1934 due to the more favorable soil and climatic conditions in the Valley during that drouth year.

The general distribution of all wheat has adhered to fairly constant pattern in the last four censuses, 1939, 1944, 1949, and 1954. In fact this trend toward constant pattern really began in 1924 and 1929.

TABLE II.—Percentage Distribution of Wheat Acres in North Dakota by Economic Areas Compiled by H. L. Walster, NDAC— Institute for Regional Research

Census Year 1889 1899 1909 1919 1924	Area 1 0.67 2.07 6.45 12.78 17.31 20.82	Area 2a 1.00 0.33 8.05 11.68 12.80 13.28	Area 3a 12.46 17.26 32.79 26.40 28.01 28.32	Area 2b 2.57 3.49 6.37 8.41 10.39	Area 3b 16.83 20.33 20.43 17.18 15.51	Area 3c 14.98 13.82 8.66 8.53 4.81	Area 4 51.06 46.62 17.26 15.09 11.01 8.33
1929	20.82	13.28	28.32	10.73	14.16	4.30	8.33
1934	15.18	4.06	28.79	3.08	19.96	1.98	26.91
1939 1944	$18.20 \\ 20.03$	12.21 $14.14$	28.84 $26.71$	8.01 9.61	13.93 14.08	5.04 4.00	13.73 11.40
1954	21.19	13.88	27.24	8.26	13.70	3.99	11.71

TABLE III—Durum\* Acres and Production Percentage Distribution by Economic Areas Compile by H. L. Walster, NDAC—Institute for Regional Research

	Area	Area	Area	Area	Area	Area	Area
	1	2a	3a	2b	3b	3c	4
Acres	3.11	3.89	47.74	4.60	27.17	6.32	7.16
1929							
Bushels	2.53	3.53	52.49	3.57	22.99	6.45	8.45
1939	٠						
Acres	12.66	6.06	46.88	6.43	19.96	6.92	11.04
Bushels	2.58	5.25	45.32	5.83	18.79	8.75	13.46
1944							
Acres	0.31	1.60	58.44	3.56	20.97	5.04	10.08
Bushels	0.33	1.82	62.56	2.63	18.08	4.42	10.16
1949							
Acres	1.37	3.80	53.93	5.01	18.50	7.48	9.92
Bushels	0.84	4.49	57.78	3.06	14.87	6.40	12.56

#### 1954

Acres	0.35	0.86	70.78	0.74	11.33	6.37	9.58
Bushels	0.74	1.41	64.28	0.84	10.68	7.12	14.93

<sup>\*</sup>Durum acreage and production was included in all spring wheat in 1889, 1899, 1909, 1919, 1924, and 1934.

# The Durum Story in 1954

Durum acres (harvested) fell to a low proportion of the total wheat acreage in 1954. Table IV sums up the status by economic areas.

Economic Areas	All Wheat Acres	Durum Acres	Percentage that Durum was of Total Wheat Acres	Other Spring Wheat Acres	Percentage that Other Spring was of Total Wheat Acres
1	1,606,715	4,022	0.25	1,595,964	99.33
2a	1,052,555	9,956	0.95	1,039,700	98.78
3a	2,065,926	819,858	39.68	1,202,848	58.22
<b>2</b> b	626,601	8,552	1.36	617,773	98.59
3b	1,039,540	131,193	12.62	905,751	87.13
3c	302,733	73,836	24.39	227,701	75.22
4	888,600	110,938	12. <del>4</del> 8	774,943	87.21

The two percentages do not add up to 100% because the all wheat figures contain a small percentage of winter wheat.

# The Total Wheat Production Story by Census Years (Bushels) and Distribution

#### by Class of Wheat Winter Year Total Durum "Other Spring" 1889 26,403,365 1899 59,888,810 1909 116,781,886 115,487,143\* 1,294,743 61,379,904\* 1919 61,540,404 160,500 1924 123,010,149 433,324 37,255,157 57,885,927 1929 95,574,408 18,014,885 17,832,954\* 181,931 1934 1939 69,261,384 24,719,976 44,421,856 119,454 152,620,442 25,722,615 32,355,699 126,849,935 47,892 1944 72,912,920 105,579,644 311,025 1949 64.543.608 3.846.988 60.548.941 147,679 1954

# A DEVICE FOR THE EXPERIMENTAL ANALOGOUS SOULTION OF CERTAIN HEAT TRANSFER PROBLEMS

# by Charles M. Harman

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Many problems in the field of heat transfer do not because of their mathematical nature readily yield to the entirely analytical approach. Among these problems, however, are a cateogry which can be facilely solved by experimental analogous means. It is this group of problems with which we here deal.

<sup>\*</sup>Includes durum.

In order to accomplish this logically we must first specifically point out the type problem to be dealt with, second establish a mathematically sound basis upon which to develop the analogy, and then finally describe a device and procedure for actually using the analogy for problem solutions.

This effort is directed toward the solution of only a particular category of problem in the general field of heat transfer. Only problems of pure conduction through homogeneous solids of known constant conductivity are considered. Further, all terminal surface temperatures must be known and no surface temperature gradients greater than those normal to the surface must occur. Only the steady state condition is allowed for and all heat flow must be considered to take place from the higher temperature surface to the lower temperature surface at a direction normal in at least one plane to the surfaces at the surfaces. Otherwise stated, the two-dimensional case is considered or the three dimensional case with one dimension constant.

Now, we must demonstrate the theoretical and mathematical justification for the analogy which we intend to use.

First, a few words about the theory of analogy itself. If we consider a number of distinct physical phenomena and if we represent these phenomena by their characteristic analytical relationships we would have a number of mathematical relationships, all functions of certain independent variables. If these relationships are identical in the formal mathematical sense, then the various phenomena are also identical from the point of view of their physical behavior. Such phenomena are said to analogous and in the case of such analogous physical phenomena it can be seen that it is possible to predict from the behavior of a given phenomena, that of a less well understood or easily measured phenomena with full confidence in the results.

In the ordinary potential-field problems to be considered here, the mathematical relationships are characteristic differential equations which stem from the physical phenomena. They are each satisfied by their own particular potential function. We will make use of the analogy between the field of thermal conduction and that of electrical conduction.

Let us first consider heat conduction. In order to be rigorous we investigate the most general expression of the heat conduction equation and cancel those terms which do not apply under our problem conditions. When this is done our equation reduces to:  $d^2t$ 

 $\frac{d^2t}{dx^2} + \frac{d^2t}{dy^2} = 0$  where t refers to temperature and x and y are orthogonal field coordinates.

An electrical field will now be considered. Suffice to say that the electrical potential e in an electrically conducting two-dimensional region of uniform electrical conductance can be represented by

$$\frac{d^2e}{dx^2} + \frac{d^2e}{dy^2} = 0 \text{ in the steady state.}$$

As can be seen, both the potentials e and t satisfy the mathematical relationship known as a simplified Laplace equation. Thus a steady electric potential field e(x,y) can be regarded as the analogue of a steady temperature field t(x,y) where equipotential lines in the voltage field correspond to isothermal lines in the temperature field. Also, orthogonal lines of electric current correspond to paths of heat flow.

At this point the Laplace equation can be futher operated on to reduce it to a more functional form. For the case of heat transfer, the Laplace equation can be reduced by various mathematical man-

ipulations to 
$$Q = -k\Delta t \int \frac{d\theta}{dx} dA$$

where Q is the heat transfered, k is the thermal conductivity, and  $\Delta t$  is the absolute difference between the inner and outer terminal temperatures. The integral depends only on the shape of the body through which heat is being conducted and is independent of the temperatures or the nature of the materials conducting the heat. It has dimensions of length and is known as Langmuir's shape factor. The difficulty of obtaining the general case solution of the integral representing the shape factor is the reason for using an experimental approach.

However some geometric shapes do yield to an analytic approach. The shape factor is in essence the ratio of the mean area to the mean length of heat transfer path. Therefore it is evident that for the case of a body with parallel faces and perfectly insulated ends the shape factor would be the ratio of the area of the faces to the perpendicular distance between them. Most shapes yield much less easily to the analytic approach and special mathematical procedures such as conformal mapping are needed.

At this point we summarize the mathematical relationships that are to be used. The equation for heat transfer can be written Q=-k Shape Factor)  $\varDelta t$  by substituting the expression "Shape Factor" for the integral. The relationships in the electrical potential field can be operated on in an exactly similar manner and can be reduced to  $I=-k_e$  (Shape Factor)  $\varDelta e$ , where I is the current,  $k_e$  is the electrical conductance and  $\varDelta e$  is the terminal voltage across the field.

Now the experimental method of shape factor determination will be investigated. If an electric circuit contains two resistances in series, the voltage drop across each will be proportional to their resistances. If the only difference between the resistances is in the configuration, then those resistances are analogous to shape factors and the following relationship can be used (Shape Factor  ${}_{1}\mathcal{L}_{e^{1}} = \mathrm{Shape}\ \mathrm{Factor}\ {}_{2}\mathcal{L}_{e_{2}}$ . If the value of (Shape Factor), is known then it is shown that any (Shape Factor)<sub>2</sub> can be determined within the

shape factor can be found by comparing it to a known shape factor.

Since the field being tested is one of uniform electrical conductance we can also obtain information regarding the field potential at any point and thereby obtain the temperature at any point in the analogous thermal field. Thus, isothermals in the temperature field can be mapped. In the case of a symmetric figure, the potentials at the position of the terminal isothermals could be moved to the lines of symmetry and the same field mapping procedure followed. The lines of symmetry must be used because they are the only known adiabatics. Electrical equipotential lines in this case would be analogous to adiabatic lines. By using both terminal isothermal and adiabatic lines as the electrical bus bars, the entire region can be mapped to show isothermals and orthogonal paths of heat flow.

Having briefly indicated the theoretical basis for the experimental analogous solution of the previously defined category of heat transfer problem, it is proper to consider next the vehicle for actually carrying out the solutions. Attention is given to keeping the device as simple as possible and to reducing the necessity of using precision instrumentation as much as possible. Accordingly, all measurements of a precise nature are made using a precision potentiometer solely. The device itself is of integral construction and contains a long-life internal power source. The experimental shapes are made of a special paper which can of course be cut to any shape. The paper is continuously metallized to provide uniform electrical conductance over its surface. The terminal equipotentials are painted on with a silver conducting paint. All resistors used for circuit voltage adjustment were precision cut from the same type paper as used for the experimental shapes.

In order to scientifically utilize the experimental analogical method it is necessary to undertake a careful analysis of the errors which such methods cause. Therefore, each source of experimental error is included, as is the posible magnitude of such error, and the means to limit such error to the smallest possible amount for operation of the device either as a director shape factor computer or as a field mapper.

The basic considerations involved in analyzing the accuracy of this method are in the materials used to simulate the test shapes and in the instrumentation.

The materials used inthe simulation consist solely of conducting paper and silver conducting paint. The conducting paint is finely divided pure silver particles dispersed in an acetone base and the paper is of the commercially available continuously conducting type.

First considering the paper, the unit resistance has frequently been found to vary as much as 10% locally and in different directions. Conductivity tests carried out on samples used here indicated somewhat better results. It has been found that while there might be con-

siderable variation percentagewise on a very local basis, this variation was reduced when larger areas were considered. The magnitude of variation was perhaps substantially unchanged but the percent variation was reduced roughly in proportion to the increase in size of the sheet being tested. As a result of these investigations it is recommended that test sections should be to as large a scale as is convenient.

The conductance of the paper seems to be slightly affected by temperature. This has no effect on the accuracy of the device since all the paper would generally be at the same temperature and although the resistance might be changed, the relative resistance which is the item measured would be unaffected. Perhaps it should be mentioned that the effect of heating or possible burning of the paper, due to the electrical energy dissipation that the paper would have to provide, has been calculated at 0.00045 watts per square inch, which could have no deleterious effect. It was found that aging has no effect in one year's time.

Next we consider the paint. The use of long bus bars painted on with the silver conducting paint may present a problem since the resistance of a long strip may become appreciable. For this reason it is recommended that soft uninsulated wire be painted onto the silver bus whenever there is a possibility or indication of this happening. A check can easily be made to determine that all of each bus bar is maintained at the same potential as the bus bar terminals of the computer. If these precautions are followed the only other source of possible error is at the electrical bond between the paint and the paper which has been tested and found to be excellent.

Having covered the source of error due to materials, the instrumentation is to be analyed to complete the analysis. Considering first the measuring instrument, it is evident that no effect on the accuracy of the results can be produced other than inaccuracy due to insensitivity if the measuring instrument at the time of reading has no current flow and must indicate a null value. This is the case since the test shape factor and the known shape factor are connected in such a manner that there is a null voltage in the instrumentation circuit when they are equal and also the entire instrumentation circuitry at the time of reading has no current flow and thus cannot influence the readings. The potentiometer has excellent sensitivity with the field density of the paper under conditions of normal battery operation.

The test shape factor must be compared to some known shape factor. This known shape factor is a pair of non-concentric circles. If the eccentricity and the size of the two circles is known, the shape shape factor can be computed from purely analytical considerations for the geometric arrangement of one circle internal to the other. The arrangement used allows for adjustment of the eccentricity and

the corresponding shape factors are available. A set of matched resistors is also necessary to divide the voltage for field mapping. These are made out of conducting paper and the deviation from the mean resistance is less than 0.6%. In field mapping, the error in distance between isothermals is proportional to this variation in internal resistances which is decidedly less than variation in test paper resistance.

In the direct shape factor comparison method the same resistors are used but in addition the non-concentric cylinder with adjustable cylinder eccentricity is involved. Here the necessity of obtaining complete electrical contact between the adjustable cylinder and the paper causes considerable difficulty. The presence of foreign particles or the physical wearing of the paper could cause poor readings. Also, tight contact of the entire cylinder face is required for complete electrical contact. Since this arrangement uses conducting paper, its variation could again cause inaccuracies. The nature of this method of approach to a problem solution is such that technique greatly adumbrates theoretical considerations in obtaining accurate results.

It is felt however that in determining shape factors by either field mapping or direct shape factor comparison an accuracy of within 10% can be expected if careful painstaking technique is followed. It is further felt that this degree of accuracy is sufficient for ordinary heat transfer computations inasmuch as the thermal conductivity values for many materials vary by this amount. More accurate conducting papers will be necessary for more accurate solutions by this method since virtually all inaccuracy is a result of variable paper resistance or poor electrical contact to the paper.

In a discussion of the accuracy of this method it would be pertinent to mention certain other types of electrical devices used for experimental analogous solution of the same type of heat transfer problems. These consist chiefly of network analyzers and liquid models. Network analyzers are much more complex both in operation and design. A continuous field is not simulated but rather a series of lumped subdivisions is developed as in the numerical relaxation method and therefore the solution has the same errors due to lumped sums and non-zero residuals as in this method. Accuracy is proportional to fineness of network mesh. This system has the advantage that large surface gradients can be handled. The liquid models are very similar in approach to the conducting sheet type. The equipment for producing the outline shape is more tedious to prepare and the inaccuracies of the conducting paper are to a certain extent comparable to a liquid model because of electrolite non-uniformity and deposition.

The method of solution of a simple example problems will now be undertaken in order to illustrate the actual solutions of problems using the analog computer. The problem is as follows: determine the heat transferred from a pipe to the surroundings. The pipe surface temperature is 220°F and the outer surface of the insulating material is at a temperature of 60°F. The conductivity of the insulating material is 0.5 British thermal units per square foot-hour-degree Fahrenheit per foot of thickness. The pipe length under consideration is 100 feet and the crossection of the insulation is one foot square externally and six inches in diameter concentric internally.

The problem is solved as follows: (1) Conducting paper is cut to simulate the crossection of the insulating material. This may be done to any convenient scale. (2) Silver conducting paint is applied at the inner and outer surfaces. A border should be left for this purpose. (3) The test section is now connected to the computer and adjustment is made until the internal shape factor exactly balances or is the same as the test shape factor. The potentiometer indicates null voltage when this is true. (4) The shape factor can now be obtained from the computer readings after multiplying by the length. The shape factor is 906 feet in this case. The heat transfered can now be calculated using Q = -k (Shape Factor)  $\Delta t$  which in this problem is Q = (-0.5) (906) (180) or Q = -81,540 Btu. per hour.

By use of the mapping stylus, the temperature anywhere in the insulating material could be closely approximated.

This problem was an extremely simple example of the type problem easily solved by this method. It should be pointed out however that an analytic approach would be most difficult or tedious nevertheless. Situations of non-isothermal terminal boundaries can also be treated, as can two or more conducting media even though their conductivity differs. Illustrations of problems of this type are beyond the scope of this paper however.

It has been the aim of the author to present a comprehensive coverage of a particular technique in the experimental analogical solution of certain problems in the field of heat transfer. The mathematical theory of the thermal-electrical analogy was investigated and the accuracy of a means of employing this analogy were discussed. The device, an analog computer, is found capable of performing its function with accuracy comparable with that ordinarily obtainable in the type problem solutions for which it was designed. The computer makes possible the solution of complicated problems in its field in a manner which is comparatively very simple and quick, and its operation is readily mastered by those familiar with the concepts of heat transfer.

# STUDIES ON PROTEIN SYNTHESIS

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### ABSTRACT

Studies have been made on the synthesis of protein using the extra-cellular amylase of a **Streptomyces sp.** as the tool. Succinategrown cells, completely devoid of amylase, were exposed to starch along with a wide variety of chemical compounds that stimulate or inhibit amylase (protein) formation.

Inhibitors of metabolic energy-yielding reactions such as azide, arsenate, and dinitrophenol stop protein synthesis at low concentrations. Several amino acids are stimulatory and reverse the inhibition of their structural analogs. Examples of pairs that give this kind of action are valine and norvaline, alanine and  $\beta$ -alanine, glutamic acid and methionine sulfoxide.

A number of purines and pyrimidines have also been found to stimulate enzyme synthesis and to reverse analog inhibition in a manner similar to the results with amino acids. The pairs of compounds investigated are: adenine and 8-azaguanine, cytosine and 2-thiocytosine, adinine and benzimidazole. The results with the purines and pyrimidines are believed to support the theory that nucleic acid synthesis must preceed the formation of new protein.

The effect of antibiotics on the system has also been studied. Most are inhibitory at relatively low concentration. Penicillin, in striking contrast, is stimulatory even at high concentrations.

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*Wheeler, Jeanette N. (Biology), University. 1924.

Wheeler, George C. (Biology), University. 1924.

Whitman, Warren (Botany), Agricultural College. 1950.

*Wilch, Lamont O. (Geology), N. D. Geol. Survey, Grand Forks. 1957.

Wildakes, William (Agronomy), Agricultural College. 1946.
                                                                                                                                                                                                                                                                                                                  Mandan. 1957.
       Wiidakes, William (Agronomy), Agricultural College. 1946.
Wills, Bernt L. (Geography), University. 1949.
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Witmer, Robert B. (Physics), University. 1925.

\*Yeager, Vernon L. (Anatomy), University. 1957.

\*Youngs, Nelson A. (Otolaryngology), Grand Forks, 1957.

Young, Ralph A. (Agronomy), Agricultural College. 1954.

\*Youngs, Roger W. (Chemistry), U. S. Bureau of Mines, Grand Forks. 1957.

Youngs, Vernon (Chemistry), School of Forestry. 1955.

Zubriski, J. C. (Soil Physics), Agricultural College. 1955.

\*Members elected in 1957