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PROCEEDINGS  
of the  
NORTH DAKOTA  
ACADEMY OF SCIENCE

Academy Founded December, 1908

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VOLUME III  
1949

---

**PUBLICATION COMMITTEE**

Herbert E. Freier (*Chairman*)

Rae H. Harris

Wilson M. Laird

G. A. Abbott (*Ex Officio*)

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*Published jointly by the University of North Dakota  
and the State Agricultural College.*

January, 1950

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U. N. D.  PRESS

GRAND FORKS, NORTH DAKOTA

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# NORTH DAKOTA ACADEMY OF SCIENCE

## ACTIVE MEMBERS, 1949

1. Abbott, G. A., (Chemistry), University, Charter Member.
2. Addicott, Harold B., (Geography), State Tchrs. College, Mayville.
3. Aldrich, Vernice, (Geography), 428 Belmont Road, Grand Forks.
4. Arnason, A. F., (Forestry), Commissioner, State Board of Higher Education, Bismarck, 1939.
5. Banasik, Orville, (Cereal Technology), Agricultural College, 1947.
6. Bavone, A. L., (Sanitary Engineering), Minot, 1947.
7. Beck, Lyle, (Dairying), Agricultural College, 1946.
8. Bolin, D. W., (Nutrition), Agricultural College, 1948.
9. Bolin, F. M., (Veterinary Science), Agricultural College, 1948.
10. Bosch, Wouter, (Chemistry), Agricultural College, 1948.
11. Bosch, Mrs. Christian, (Chemistry), Agricultural College, 1949.
12. Brezden, William, (Chemistry), State Mill and Elevator, Grand Forks, 1945.
13. Broberg, J. W., (Chemistry), Agricultural College, 1948.
14. Bryant, R. L., (Experiment Station), Dickinson, 1948.
15. Burr, Alexander C., (Chemical Engineering), State Research Foundation, Bismarck, 1940.
16. Chetrick, M. H., (Chemical Engineering), University, 1947.
17. Christoffers, Harry, (Chemistry), University, 1949.
18. Christoffers, Mrs. Harry (Chemistry), University, 1949.
19. Clagett, C. O., (Agr. Chemistry), Agricultural College, 1947.
20. Cooley, A. M., (Chemical Engineering), University, 1938.
21. Coon, Ernest D., (Chemistry), University, 1923.
22. Cram, Sheldon, (Physics), State Tchrs. College, Dickinson, 1948.
23. Daly, J. J., (Mining Engineering), University, 1948.
24. Davis, Joe, (Biology), State Wild Life Adm., Bismarck, 1938.
25. Dunbar, Ralph E., (Chemistry), Agricultural College, 1938.
26. Dunlap, W. A., (Public Health), Assistant Director, State Public Health Laboratory, Bismarck, 1946.
27. Ellman, Robert, (Fuels), U. S. Bureau of Mines Station, University, 1949.
28. Evanson, Harlow, (Physics), School of Forestry, Bottineau, 1947.
29. Eveleth, F. F., (Veterinary Science), Agricultural College, 1944.
30. Espe, Dwight, (Dairy Husbandry), Agricultural College, 1947.
31. Facey, Vera, (Botany), University, 1948.
32. Fallgatter, Wayne, (Fuels), U. S. Bureau of Mines station, University, 1949.
33. Fischer, R. G., (Medicine), University, 1949.
34. Flor, Harold H., (Pathology), Agricultural College, 1932.
35. Fleetwood, C. W., (Chemistry), Agricultural College, 1948.
36. Ford, Kenneth, (Experiment Station), Dickinson, 1948.
37. Fordice, Ira, (Chemistry), Agr. Supply Co., Grand Forks, 1947.

38. Frank, Richard E., (Chemistry), University, 1949.
39. French, Harley E., (Medicine), (Dean Emeritus), University, 1911.
40. French, Leslie A. (Engineering), State Highway Department, Bismarck, 1943.
41. Freier, Herbert E. (Chemistry), University, 1947.
42. Gault, Alta R., (Physiology), University, 1949.
43. Gillette, John M., (Rural Sociology), University, Charter Member.
44. Goldsby, Alice, (Veterinary Science), Agricultural College, 1946.
45. Grimes, Ruby, (Mathematics), Agricultural College, 1946.
46. Grossman, Irving, (Geology), University, 1947.
47. Gustafson, Ben G., (Chemistry), University, 1939.
48. Hagen, Irvin, (State Seed Dept.), Agricultural College, 1946.
49. Hankinson, Kermit, (Mathematics), Agricultural College, 1946.
50. Hanning, Irene (Mrs. Irene Podoll) (Mathematics), 618 Third St., Bismarck, 1945.
51. Harrington, L. C. (Engineering), University, 1913.
52. Harris, Rae H., (Cereal Technology), Agricultural College, 1946.
53. Hart, Harry B., (Chemistry), Jamestown College, Jamestown.
54. Helgeson, E. A., (Botany), Agricultural College, 1936.
55. Hemphill, Perry V., (Agricultural Extension), Agricultural College, 1937.
56. Henderson, Donald, (Physics), University, 1945.
57. Hill, Glenn A., (Mathematics), Agricultural College, 1946.
58. Hoepfner, Jerome, (Fuels), U. S. Bureau of Mines station, University, 1949.
59. Holtz, John C., (Fuels), U. S. Bur. Mines station, University, 1949.
60. Hundley, John L., (Physics), University, 1930.
61. Hurst, H. J., (Physics), State School of Forestry, Bottineau, 1942.
62. Jensen, C., (Dairy Husbandry), Agricultural College, 1927.
63. Johanson, J. F., (Social Science), Carrington, 1939.
64. Johnsgard, Gordon, (Soils), Agricultural College, 1947.
65. Johnston, Jr., James W., (Zoology), Agricultural College, 1948.
66. Jones, James R., (Geology), University, 1949.
67. Jones, Mrs. James R., (Geology), University, 1949.
68. Kamps, Theodore, (Fuels), U. S. Bureau of Mines station, University, 1949.
69. Kelly, Eunice, (Nutrition), Agricultural College, 1944.
70. Kirk, H. H., (Biology), Supt. of Schools, Fargo, 1938.
71. Kjerstad, C. L., (Psychology), University, 1937.
72. Kling, Robert R., (Medicine), University, 1949.
73. Klosterman, Earl W., (Animal Nutrition), Agricultural College 1948.
74. Klosterman, Harold, (Chemistry), Agricultural College, 1948.

75. Knoll, Clement, (Engineering), State School of Forestry, Bottineau, 1949.
76. Kohanowski, Nicholas, (Geology), University, 1949.
77. Koons, Melvin E., (Public Health Laboratory), University, 1943.
78. Koth, Arthur W., (Mining Engineering), University, 1939.
79. Kruschwitz, Earl W., (Physics), State Teachers College, Valley City, 1947.
80. Kube, Wayne, (Fuels), U. S. Bur. Mines station, University, 1949.
81. Laird, Wilson M., (Geologist), University, 1941.
82. Larson, Edith, (Biology), University, 1947.
83. Larson, Kermit, (Chemistry), (On leave, Grad. Student, University of California), 1943.
84. Lauster, K. C., (State Health Dept.), Bismarck, 1944.
85. Lejeune, A. J., (Agronomy), Agricultural College, 1947.
86. Leraas, Marvin A. (Agronomy), Agricultural College, 1947.
87. Lundy, John L., (Anesthesiology), Mayo Clinic, Rochester, Minnesota, 1940.
88. McMillan, William, (Chemistry), State Research Foundation, Bismarck, 1947.
89. McKean, William, (Animal Biology), Agricultural College, 1947.
90. Marwin, Richard M., (Bacteriology), University, 1949.
91. Maher, George, (Agricultural Chemistry), Agricultural College, 1948.
92. Mason, Sewell, (Mathematics), University, 1946.
93. Mattson, Harold, (Horticulture), Agricultural College, 1937.
94. Miller, Cap E., (Agricultural Economics), Agricultural College, 1919.
95. Miller, C. E., (Pharmacy), Agricultural College, 1947.
96. Moberg, Wenzel, (Geography), Agricultural College, 1943.
97. Moomaw, Leroy, (Experiment Station), Dickinson, 1943.
98. Moran, Walter H., (Chemistry), University, 1928.
99. Moore, C. C., (Chemistry), Agricultural College, 1948.
100. Munro, J. Alex, (Entomology), Agricultural College, 1927.
101. Murphy, H. E., (Chemistry), State Teachers College, Dickinson.
102. Nelson, Caspar I., (Bacteriology), Agricultural College, 1915.
103. Norum, Enoch, (Soils) Agricultural College, 1948.
104. Nystuen, Peter, (Agriculture), Agricultural College, 1947.
105. Oehler, Mrs. Alma, (Nutrition), State Mill and Elevator, Grand Forks, 1945.
106. Ongstad, Orvin C., (Fuels), U. S. Bureau of Mines station, University, 1949.
107. Oppelt, W. H., (Fuels), U. S. Bur. Mines station, University, 1949.
108. Opton, Edward, (Physiology), Agricultural College, 1946.
109. Overby, G. O., (Physics), State Tchrs. College, Valley City, 1947.
110. Overn, A. V., (General Science), University, 1949.

111. Owens, Paul, (Owens Floral), Grand Forks, 1945.
112. Posin, D. Q., (Physics), Agricultural College, 1948.
113. Post, R. L., (Entomology), Agricultural College, 1948.
114. Potter, Loren, (Botany), Agricultural College, 1948.
115. Potter, W. F., (Medicine), Dean of Med. School, University, 1949.
116. Promersberger, W. J., (Agricultural Engineering), Agricultural College, 1938.
117. Reid, Russell, (Natural Science), State Museum, Bismarck, 1940.
118. Reiersen, W. T., (Biology), State Normal and Industrial School, Ellendale, 1940.
119. Rhodes, L. D., (Physical Science), State Teachers College, Valley City, 1920.
120. Riley, K. W., (Chemistry), City Chemist, Grand Forks, 1945.
121. Robertson, Ina G., (Geography), State Teachers College, Valley City.
122. Rognlie, Philip, (Mathematics), University, 1946.
123. Saiki, Arthur K., (Pathology), University, 1929.
124. Sands, Frederick H., (Chemistry), Agricultural College, 1946.
125. Samuelson, Theodore A., (Pharmacy), State Laboratories, Bismarck, 1943.
126. Saugstad, Stanley, (Entomology), Wild Life Adm., Bismarck, 1939.
127. Schultz, Frederick, (Physics), University, 1948.
128. Schultz, J. H., (Horticulture), Agricultural College, 1948.
129. Schlamb, Kenneth, (Experiment Station), Dickinson, 1948.
130. Sebens, Walter F., (Agriculture), Greater North Dakota Association, Fargo, 1948.
131. Severson, Donald (Chemical Engineering), University, 1949.
132. Shute, Leonard F., (Chemistry), Agricultural College, 1948.
133. Seymour, J. H., (Biology), State Teachers College, Valley City.
134. Shepperd, Mrs. Adele G., (Chemistry), 1018 Seventh Street North, Fargo, 1911.
135. Shepperd, Wyman (Horticulture), East Grand Forks, Minn., 1943.
136. Sibbitt, L. D., (Cereal Technology), Agricultural College, 1946.
137. Sinner, Eugene M., (Pharmacy), State Laboratories, Bismarck, 1945.
138. Smith, Glenn, (Experiment Station), Agricultural College, 1930.
139. Smith, Grant W., (Chemistry), University, 1948.
140. Stafne, Gilbert J., (Farm Bureau), Fargo, 1948.
141. Staley, R. C., (Mathematics), University, 1946.
142. Stebner, Jack, (State Laboratories), Bismarck, 1943.
143. Steinmeier, L. D., (Chemistry), State School of Forestry, Bottineau, 1947.
144. Stevens, O. A., (Botany), Agricultural College, 1910.

145. Stewart, Donald L., (Chemistry), American Beet Sugar Co., East Grand Forks, Minnesota, 1943.
146. Sudro, W. F., (Pharmacy), Agricultural College, 1911.
147. Svore, Jerome, (Sanitary Engineering), State Health Dept., Bismarck, 1943.
148. Taintor, E. J. (Agriculture), Taintor Seed House, Grand Forks, 1945.
149. Thompson, John C., (Mathematics), State Teachers College, Dickinson, 1948.
150. Thompson, Matilda, (Mathematics), Agricultural College, 1946.
151. Treumann, William B., (Chemistry), Agricultural College, 1947.
152. Tuneberg, Everett H., (Chemistry), State School of Forestry, Bottineau, 1949.
153. Van Heuvelen, W., (Chemistry), State Health Dept., Bismarck, 1945.
154. Voedisch, Fred W., (Geology), Grand Forks, 1935.
155. Waldron, L. R., (Agronomy), Agricultural College, 1910.
156. Walster, H. L., (Director Experiment Station), Agricultural College, 1920.
157. Wanner, Donald F., (Agricultural Economics), Farm Security Administration, Jamestown, 1947.
158. Wardner, Arthur K., (Agricultural Chemistry), Flaot Farms Co., Grand Forks, 1938.
159. Weeks, Oliver, (Bacteriology), Agricultural College, 1947.
160. Wendland, Ray T., (Chemistry), Agricultural College, 1948.
161. Weyer, Albert E., (Chemistry), Agricultural College, 1948.
162. Whedon, Arthur D., (Zoology), Agricultural College.
163. Wheeler, George C., (Zoology), University, 1924.
164. Wiidikas, William, (Agronomy), Agricultural College.
165. Wills, Bert L., (Geography), University, 1949.
166. Winchester, Burl, (Animal Husbandry), Agricultural College, 1947.
167. Witmer, R. B., (Physics), University, 1925.
168. Witz, R. L., (Agricultural Engineering), Agricultural College, 1947.



## NORTH DAKOTA ACADEMY OF SCIENCE

*Minutes of the Forty-first Annual Meeting**University, Grand Forks, North Dakota***MAY 6 and 7, 1949**

The Academy was called to order at 9:15 a. m., May 6th, in Room 18, Merrifield hall on the University campus by Vice President Dr. Ralph E. Dunbar, in the absence of President Dean R. B. Witmer, who was attending a conference of Deans at Oxford, Mississippi.

After extending a welcome to members and visitors, the acting president introduced the program of papers. The seven papers scheduled for the morning session were read and briefly discussed.

At 12:00 o'clock, luncheon was held at the University Commons, complimentary to the visitors, with local members lunching with their guests.

The afternoon session was called at 1:35 p. m. in Room 18, Merrifield hall. Dr. Dunbar announced the appointment of the following committees: Committee on Resolutions: Harry B. Hart, Chairman; H. A. Robinson, Wouter Bosch. Committee on Nominations: Rae H. Harris, Chairman; A. M. Cooley, J. Alex Munro.

The twelve papers listed for the afternoon program were all read and brief discussions followed. Average attendance was about 60. The session adjourned at 4:45 p. m.

The Academy Dinner was held at 6:00 p. m. in the large dining hall of the University Commons. The occasion was unique, in that it was a joint dinner meeting with the recently organized Red River Valley Section of the American Chemical Society, and the dinner was sponsored by the local Chapter of Sigma Xi.

As president of Sigma Xi, Professor A. M. Cooley extended the warm welcome of the host society, and also that of President John C. West, of the University, whose duties compelled him to be absent, to the members of the Academy and of the Section and their guests.

He then introduced Mr. Richard Pearson, a graduate student in Chemistry at the University, and junior member of the American Chemical Society, who sang two very pleasing baritone solos, accompanied by Miss Mary Sussex, pianist.

Mr. Cooley then presented the gavel to Acting President Dunbar of the Academy, who extended the felicitations of the Academy to the Section of the American Chemical Society, and called upon Dr. G. A. Abbott, Chairman of the Section, to introduce the distinguished guest speakers who were sponsored by the Section.

They were Dr. Jean Felix Piccard, famous for his investigations of the stratosphere, and his talented wife who participated in the program with her husband. Mrs. Piccard is also a trained scientist and a woman of charming personality. She is a licensed balloon pilot and served as pilot for her husband in their stratosphere ascensions. Dr. Piccard is Professor of Aeronautical Engineering at the University of Minnesota.

In his address, Dr. Piccard gave a clear summation of present knowledge of the stratosphere, and explained why further investigations may reveal facts of great practical as well as theoretical interest. His balloon carried him to a height of 50,000 feet, which he said is about the maximum which can be attained with a single balloon. This is due to the fact that in increasing the size of a balloon the weight increases out of proportion to the increased lifting power. This suggested to him the use of smaller balloons in groups or clusters arranged in tandem after the manner of the Eskimo dog teams. Many of his colleagues ridiculed the idea, and warned him that his balloon teams would get into dangerous "dog fights." But experimental flights with such clusters proved entirely satisfactory, and the speaker stated with enthusiasm that he was prepared to ascend to a height of 100,000 feet, if and when funds were made available for such an attempt.

Mrs. Piccard followed her husband on the program, delighting her audience with her vivid accounts of her experiences, and particularly by her simplicity and the warmth of her human interest.

Following the addresses, moving pictures of the famous stratosphere flight at Detroit were shown, as well as the experimental flights testing the use of the balloon clusters. The latter were very beautiful. The large clusters of five foot balloons, made of shining plastic resembled great bunches of grapes, or clusters of pearls as they carried the spherical gondola high into the clouds. The audience was also interested to note that Mrs. Piccard was the pilot in charge, sitting on the top of the gondola and giving commands to the ground crew, finally descending into the gondola only after the balloon was free and rising rapidly.

Following the formal program, the guest speakers graciously remained to answer the many questions asked by members of the enthusiastic audience. The attendance at dinner was 113.

### **Saturday, May 7, Room 18, Merrifield Hall**

Assembly call, at 9:30 a. m., Dr. Dunbar presiding.

The five scheduled papers were read and discussed.

After a brief recess, the Academy was called to order for the Business Session.

The minutes of the 40th annual meeting were read by the Secretary and approved.

The report of the Treasurer was presented, and accepted by unanimous vote.

Reporting for the Committee on Publication, chairman Wilson M. Laird announced the publication and distribution of the second volume of the Proceedings of the Academy. On account of pressing duties, he asked to be relieved of further service on the Committee. After some discussion, it was voted to accept his resignation from the chairmanship; but to request him to remain on the committee in an advisory capacity. Upon motion of Dr. Laird, second, Dr. Rae H. Harris, Dr. Herbert E. Freier, was unanimously elected to replace Dr. Laird as chairman of the Committee. Dr. Alex J. Munro also asked to be relieved of service on the Committee, and his resignation was reluctantly accepted. The membership of the Committee is as follows: Herbert E. Freier, Chairman, Rae H. Harris, Wilson M. Laird, and (ex officio) Secretary-Treasurer, G. A. Abbott.

J. Alex Munro, representative in the Council of the A. A. A. S. had no formal report, as he was unable to attend the meeting of the A. A. A. S.

Other members were called upon for informal reports on other national scientific meetings attended. Brief reports were made by Wilson M. Laird, on the meeting of geologists, at Denver, and by Grant W. Smith, who attended the meeting of the American Chemical Society, in San Francisco.

The report of the Committee on Resolutions was presented by Chairman H. B. Hart, and adopted, on motion of G. A. Abbott, second, Robert Varland. Reporting for the Committee on Nominations, Chairman Rae H. Harris presented nominations for the officers and members of the Executive Committee. In the absence of further nominations, the unanimous ballot was cast for the following:

President - - Dr. Ralph E. Dunbar, Agricultural College

Vice President Dr. A. K. Saiki, University

Secretary-Treasurer - - Dr. G. A. Abbott, University

Additional Members of the Executive Committee:

Dr. H. B. Hart - - - - Jamestown College

Prof. M. A. Leraas State Teachers College, Valley City

The following new members were elected:

1. Bosch, Mrs. Christian, (Chemistry), Agricultural College.
2. Christoffers, Harry, (Chemistry), University.
3. Christoffers, Mrs. Harry (Chemistry), University.
4. Ellman, Robert, (Fuels), U. S. Bureau of Mines station, University.
5. Fallgatter, Wayne, (Fuels), U. S. Bureau of Mines station, University.
6. Fischer, R. G., (Medicine), University.
7. Frank, Richard E., (Chemistry), University.
8. Gault, Alta R., (Physiology), University.
9. Hoepfner, Jerome (Fuels), U. S. Bureau of Mines station, University.
10. Holtz, John C., (Fuels), U. S. Bureau of Mines station, University.
11. Jones, James R., (Geology), University.
12. Jones, Mrs. James R., (Geology), University.
13. Kamps, Theodore, (Fuels), U. S. Bureau of Mines station, University.
14. Kling, Robert R., (Medicine), University.
15. Knoll, Clement (Engineering), State School of Forestry, Bottineau.
16. Kohanowski, Nicholas, (Geology), University.
17. Kube, Wayne, (Fuels), U. S. Bureau of Mines station, University.
18. Marwin, Richard M., (Bacteriology), University.
19. Ongstad, Orvin C., (Fuels), U. S. Bureau of Mines station, University.
20. Oppelt, W. H., (Fuels), U. S. Bureau of Mines station, University.
21. Overn, A. V., (General Science), University.
22. Potter, W. F., (Medicine), University.
23. Severson, Donald, (Chemical Engineering), University.
24. Tuneberg, Everett H., (Chemistry), State School of Forestry, Bottineau.
25. Wills, Bert L., (Geography), University.

The Academy adjourned at 11:45 a. m., to meet at the Agricultural College, May 5 and 6, 1950. Following adjournment, luncheon was held at the University Commons, complimentary to visiting members.

RALPH E. DUNBAR,  
Vice President (Acting Chairman)

G. A. ABBOTT,  
Secretary-Treasurer.

## DEHYDRATED POTATO SILAGE

*C. A. Wardner*

*Flatt Farms Co.*

It has been observed from past experience that the feeding of dehydrated potatoes, which consist of rejects from a dehydration plant during the past war, resulted in optimum daily gains and high quality well marbled meat.

The objects of these experiments were to determine the conditions for the fermentation of potatoes to form silage, the conditioning of the silage for economical drying and the drying characteristics of the silage in a direct heat rotary dryer.

The raw milled potatoes when mixed with alfalfa meal, cracked corn, or millet and allowed to ferment in loosely covered drums at temperatures between 40 and 70°F. formed a satisfactory silage. Raw milled potatoes with nothing added fermented satisfactorily to form silage under the physical conditions stated above.

The conclusions from this investigation were as follows:

1. Up to 46% of the original moisture content of potato pulp may be removed by draining after fermentation and the juice contained only 3.1% to 4.7% solids.
2. The addition of alfalfa meal and stems improves the drying characteristics of potato pulp and also lowers the percentage of solids found in the juice issuing from the press.
3. Under commercial operations dehydrated potato silage can be produced economically.
4. Dehydrated potato silage may constitute a major portion of the rations for fattening steers.
5. Very satisfactory gains are made when dehydrated potato silage is included in a feeding ration.

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## ZINC STIMULATION OF POTATOES

*Wm. G. Hoyman*

*Associate Plant Pathologist, North Dakota Agricultural Experiment Station and North Dakota State Seed Department.*

Potatoes are one of the many species of plants known to require zinc for normal growth. Extensive water culture experiments by Schreven<sup>1</sup> have shown that zinc is necessary for potatoes. Zinc has been included in potato fertilizers but the response in yield has not been too encouraging. Extensive investigations conducted in Minnesota by Rost, Kramer, and McCall<sup>2</sup> failed to show that zinc-containing

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<sup>1</sup> Schreven, D. A. van Zink als een noodzakelijk element voor de suikerbiet en de aardappelplant. Meded. Inst. Suikerbieten. 7, 1-26 (1937).

<sup>2</sup> Rost, C. O., Kramer, H. W., and McCall, T. M., Fertilizers for Potatoes in the Red River Valley. Minn. Agr. Exp. Sta. Bull. 385, (1945).

fertilizers increased the yield significantly. Zinc deficiency symptoms have never been reported in the Red River Valley of North Dakota on potatoes or any other crop.

Many new potato fungicides introduced during and following the last world war included some organic materials containing zinc. These and a few copper-containing fungicides have been used at three different locations in the Red River Valley of North Dakota since 1946. A triple lattice design with 16 treatments, each replicated six times, has been used each year for the testing of the various materials. Among the zinc-containing materials were Zerlate, Dithane D-14, Parzate, Dithane Z-78, Tri-Basic-Nu-Z, Copper-Zinc Chromate and Zinc Nitrodithioacetate. The dusting and spraying of these materials on the foliage has increased the yield over the check plot in years when foliage diseases were not a factor. In 1948 the increases ranged from 20 to 40 bushels per acre. Most of the yield increases were significant at the 1 per cent level.

Zinc sulphate was applied to three different plots in 1948. This chemical has little, if any, fungicidal value but the yield from each of these plots was highly significant.

During the three years' research there was no instance where the yield from a plot dusted or sprayed with a copper fungicide was significantly higher than the check. In some cases the copper-treated plots were lower in yield than the check.

#### SUMMARY

The application of zinc-containing dusts and sprays to vines showing no external symptoms of zinc deficiency increased the yield of potatoes grown in the Red River Valley of North Dakota.

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## INFLUENCE OF CROPPING PROCEDURES ON WHEAT YIELD AND QUALITY

*R. H. Harris, L. D. Sibbitt and T. E. Stoa*

*North Dakota Agricultural Experiment Station*

The data discussed in this paper have been collected from investigations covering 26 years, and carried out in the rotation and fertility plots, Department of Agronomy at the Experiment Station at Fargo, with the object of ascertaining the influence of various factors on hard red spring wheat quality and yield. The quality criteria discussed are test weight per bushel, wheat protein content and loaf volume. The experiments fall into two divisions, classified by agronomic treatments. In one, the cropping sequence and fertility plots, wheat follows clover, timothy or millet, and peas. In addition, manure is applied to one of each pair of plots treated as described, while the other member of the pair is without fertilizer application. The other experiment is a sweet clover management series, which con-

sists of seven separate tillage and cultural treatments, one of which is a control or check plot without clover, while the remaining six consist of different forms of clover management.

Sweet clover or peas increased the yield of the following wheat crop, while millet and timothy decreased the yield. This crop treatment also lowered wheat protein content and baking strength of the resultant flours. Sweet clover and peas tended to slightly improve the protein content of the next wheat crop. In continuous wheat production 12 inch drilling slightly increased yield and protein content above the 6 inch drilling, but these differences were not statistically significant. Manurial treatments improved yields and protein content only to a slight extent; the largest improvement was secured following timothy or millet. Differences between years were much larger than those between treatments.

For the sweet clover trials, treatments and year of growth had a significant effect on yield and size of loaf baked from the flour milled from the wheats. Test weight and protein content were not influenced by treatment, however. The effect of yearly variations in weather was highly important.

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## GEOMORPHOLOGY OF THE INTERIOR SALINE BASINS OF WESTERN NORTH DAKOTA (SUMMARY)

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Two years of field and laboratory work on the arid northwest has uncovered several promising sodium sulfate deposits. In addition, geologic and topographic mapping by the United States Geological Survey has resulted in the accumulation of enough evidence to make possible the formulation of some tentative hypothesis on the origin of the salt beds.

Two such hypothesis have been advanced for the North Dakota area. The first, advanced by Irving J. Witkind of the United States Geological Survey, explains the distribution of the salt deposits by postulating the existence of two types of channels. Both are filled with glacio-fluvial sediment, although one is buried beneath till. The till-buried channel is marked by a line of kettles reflecting its former course. Ground water confined under hydrostatic pressure by this layer of till reaches the surface as springs within these kettles. The ponded waters cannot drain through the impermeable till and are gradually concentrated by evaporation to form sodium sulfate lakes. Nearby fresh lakes, also associated with glacial outwash, do not become saline because of the loss of dissolved salts by subsurface drainage.

A second hypothesis, advanced by the author, holds that many of the fresh lakes lack appreciable quantities of dissolved salts because of occasional overflow to nearby basins. The proximity of saline lakes topographically lower than the adjacent fresh water lakes tends to confirm this view. Other evidence includes the presence of connecting spillways and data on ground water analyses. It is emphasized that while the preponderance of evidence favors the theory of temporary basin integration, the alternative theory may be operative, at least locally. In the absence of detailed drilling, the source material has not yet been determined with any certainty. It may be the glacial till, the underlying bedrock, or both.

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## RAW MATERIALS FOR THE MANUFACTURE OF PORTLAND CEMENT IN NORTH DAKOTA

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*North Dakota Research Foundation*

A local source of cement, based upon the state's resources, has been sought for 55 years. Pioneering investigations were made by Babcock, Budge<sup>1</sup> and others. More recently the N. Dak. Research Foundation has studied the problem<sup>2</sup>. The Foundation has concluded that the North Dakota market can support a plant with annual capacity of 400,000 barrels of cement a year. This justifies a plant if raw materials are available.

In discussing raw materials we need consider only the search for a calcareous material to provide the major source of lime. The other raw materials, (1) silica, (2) alumina, (3) coal, oil or gas, (4) water and (5) gypsum, are either readily available or required in small amounts.

### Limestone Survey

A survey of the lime deposits of the state has been completed. Over 400 samples have been analyzed and some 1,600 feet of drill hole sunk. In the northeast corner of the state lies the Niobrara formation of limestone, more popularly called "cement rock". This material, although it was used to make natural cement in the early 1900's, is of too low grade for the manufacture of Portland cement. Because of its chemical nature it cannot be up-graded by flotation, nor is there near at hand other deposits or sources of high grade lime to be used for improving the grade.

Deposits of marl (clay and carbonate of lime) occurring just south of Devils Lake have been investigated by Budge<sup>1</sup>, Burr and Laird. Although some of these deposits are high in lime, their grade is variable, and the quantity is small.

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<sup>1</sup> Budge, W., School of Mines Mineral Resources Survey of 1937. "Marl Deposits Near Devils Lake." Report of N. Dak. State Planning Board, (March 25, 1938).  
<sup>2</sup> MacMillan, W., "Is a Portland Cement Plant Technically and Economically Practical in North Dakota?" Part 1, "Can the North Dakota Market Support a Plant?" N. Dak. Research Foundation, Unpublished studies, (1949).



The most promising deposit investigated is Colgrove Butte, a part of the White River formation in southwestern N. Dak. Thus far 11 holes have been sunk in this butte. The total drilling was 353 feet with an average depth of 32 feet, although one hole was drilled to a 50 foot depth. The lime extends as far as 30 feet below the surface. In general the grade of the limestone decreases with depth, although some exceptions occur around depths of 10, 12 or 15 feet. This limestone is low grade and would require up-grading. Tests made by the Separation Process Co. of Catasqua, Pennsylvania indicate that the percent of calcium carbonate can be increased in this way.

#### Raw Material Quantities and Requirements

After eliminating some of the White River deposits due to the low grade, the quantity of material and the accessibility, we may select a few of the deposits for comparison (see Table I).

**TABLE I**

#### Limestone Deposits in Selected White River Deposits<sup>4</sup>

	Tons
A. Colgrove Butte	
1. Powers <sup>3</sup> , 2 foot layer	3,948,000
2. Thorsteinsson	
a. 80% layer, 1.4 feet thick	1,600,000
b. 54       "    3.0   "   "	5,300,000
c. 49       "    5.0   "   "	8,800,000
B. Bull Butte	
1. Powers, 2 foot layer	495,000
C. Antelope Buttes	
1. Powers	202,000
D. Lefor Area	
1. Powers	6,500,000

Knowing that the Colgrove Butte lime can be up-graded by flotation sufficiently to serve as a suitable raw material, we may calculate the quantity necessary for one barrel of cement. The available materials required for one barrel of cement are shown in Table II.

**TABLE II**

#### Summary of Materials for One Barrel of Cement<sup>4</sup>

A. Stone	530 lbs.	36.8%
B. Clay	59	4.1
C. Lignite	250	17.4
D. Water	589	40.8
E. Gypsum	11	0.8
	Total 1,439 lbs.	100.0%

<sup>3</sup> Powers, W., "Cement Rock and Limestone Deposits in North Dakota," Unpublished studies, (1946).

<sup>4</sup> "Is a Portland Cement Manufacturing Plant Technically and Economically Practical in North Dakota?" Part II, "Are Raw Materials Available in sufficient Quantity and Satisfactory Quality to Justify a Plant?" N. Dak. Research Foundation, unpublished studies, (1949).

With Table II as a guide we can calculate the life of the Colgrove Butte deposit in supplying the raw materials for 400,000 barrels of cement a year. It should be noted that the thicker the layer used, the longer the life of the deposit but the lower the per cent of calcium carbonate and therefore the higher the operating costs. Table III shows the estimated life of the Colgrove Butte deposit.

**TABLE III**  
**Estimated Life of the Colgrove Butte Deposit<sup>4</sup>**

A. 80% deposit	14.5 years
B. 54% deposit	32.4 years
C. 49% deposit	48.8 years

### Cost Estimates

A study of published data on the cost of cement plants together with information supplied by two concerns with wide experience in building cement plants has led to the assumption that the initial

**TABLE IV**  
**Estimated Total Operating Expenses<sup>5</sup>**

<b>Fixed Charges</b>		<b>Per yr.</b>	<b>Per bbl.</b>
Depreciation or amortization (20 yrs.)	5%		
Interest	6		
General Maintenance	3		
Insurance	2		
Taxes	3		
	19%	\$685,000	\$1.71
<b>Operating Charges</b>			
Stone	13.2 cents/bbl.		
Clay	1.6		
Lignite	18.7		
Gypsum	2.0		
Oil and chemicals	1.0		
Water	0.5		
	37.0 cents/bbl.	\$148,000	\$0.37
Power		80,000	0.20
Labor		160,000	0.40
Salaries		60,000	0.15
<b>Social Taxes</b>			
Social Security (160,000+30,000) (0.02) =	\$3,800		
Workmens Compensation 130,000 at 1%			
30,000 at 5%	2,800	6,600	0.01
<b>TOTAL</b>		\$1,139,600	\$2.84

<sup>5</sup> Ibid. Part III, "Estimated Investment and Operating Costs."

investment, exclusive of operating capital, will be \$3,000 per barrel of daily capacity. On a 1,200 barrel per day plant, this investment would be \$3,600,000.

For a preliminary estimation the total cost of operation is assumed to be the sum of three terms: (A) fixed charges, (B) operating charges, (C) social taxes. Table IV sums up these costs.

### Conclusions

1. The Niobrara rock cannot be used for cement without addition of a high grade limestone, which is not available within economic distance.

2. The marl deposits are too small in size.

3. Indications are that Portland cement could be made with up-graded White River limestone from Colgrove Butte in conjunction with clay. However, final decision cannot be made on the basis of this preliminary study. It would be necessary to drill the Colgrove Butte deposit more thoroughly and to examine the Lefor area more closely.

4. The figures on cost are purposely high and revision will be downward. Estimated cost of operation compares not too unfavorably at this state of investigation with those reported or estimated for plants now in operation.

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## SULFUR IN NORTH DAKOTA LIGNITE

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Although sulfur is a minor constituent of lignite, it is an important one. The study of sulfur in North Dakota lignite has received little attention, but it is worthy of consideration because reserves of low-sulfur coal are being steadily depleted. The amount of sulfur in coal is used in indicating the value of a coal for use under steam boilers, in the manufacturing of coke and illuminating gas, and in ceramic industries.

Sulfur is an undesirable element in coal for several reasons. The products of combustion and decomposition of the sulfur compounds flowing out of the stacks pollute the air making it unpleasant and irritating; causing destruction to stone and concrete buildings, to paint, and to laundry. The sulfur compounds are corrosive, too, and may cause rapid deterioration to all metallic parts with which they come in contact. Sulfur is also objectionable in gas synthesis. The formation of clinkers in the fuel bed is one of the most serious troubles the coal consumer fears with high-sulfur coal. Another objection to such a coal is that the slow oxidation of the iron sulfide lowers the heating value of the coal and increases the danger of spontaneous combustion.

Sulfur constituents in lignite consist of (1) intrinsic impurities that are derived from the original vegetable material or by sedi-

mentation or precipitation during accumulation of coal-forming vegetation and (2) extrinsic impurities caused by infiltration of hydrogen sulfide waters and periodic deposition. The main forms in which sulfur exists in North Dakota lignite are: iron and calcium sulfate, iron pyrite, and organic compounds. Sulfate sulfur is almost absent in freshly mined coal. Iron pyrites occur in form of balls, lenses, nodules, continuous layers, thin sheets or flakes, and in very fine microscopic particles. Next to pyritic sulfur the most prominent form in coal is that in organic combination with the coal substance.

In studying the origin of sulfur in lignite, it is found that proteins are of great importance in the history and chemistry of the sulfur in coal. Proteins are relatively easily decomposed, yet a considerable amount of sulfur may have been retained in the original tissue as organic sulfur. This would be indicated by the fact that a large amount of vegetable matter escapes putrefaction, as in peat formation and in the large amount of well preserved plant tissue found in lignites. The inorganic sulfur is almost entirely combined as iron pyrites and the infiltration of hydrogen-sulfide waters is doubtless a factor in these combinations. It appears that the percentage of sulfur in lignite is controlled by the conditions existing during the laying down of the bed.

There are three methods accepted by the American Society for Testing Materials for determining total sulfur in coal. The one given in Bureau of Mines Technical Paper 8 is the Eschka method, which consists of heating the coal with magnesium oxide and anhydrous sodium carbonate. Another method is the Bomb-washing in which the sulfur is determined in the washings from the oxygen bomb calorimetric determination. The third method makes use of the combustion of coal by means of sodium peroxide in a sulfur bomb.

For certain purposes it is desirable to know how the total sulfur is combined in the coal and the exact amount of each form present. An accurate analytical method for the systematic analysis of sulfur forms in coal has been developed by Powell and Parr. A dilute hydrochloric acid extract is analyzed for iron and sulfur, the sulfur representing the sulfate sulfur. The analysis of the extract from dilute nitric acid gives the iron and sulfur as pyrites plus the sulfates. The latter form has already been determined, so by subtraction, the pyritic sulfur content of the coal is given. This may be checked for pyritic sulfur by the following calculation: the hydrochloric acid soluble iron is subtracted from the nitric acid iron, thereby giving the iron combined as pyrites.

Total organic sulfur can be calculated by subtracting the percentage of sulfate and pyritic sulfur from the percentage of total sulfur. The phenol soluble type of organic sulfur can be calculated from the difference between the total sulfur content of the coal and the sulfur content of the coal residue after phenol extraction. The "humus"

type of organic sulfur is not extracted by phenol. The total organic sulfur is determined directly by the Eschka method on the residues from dilute nitric acid extraction, from which the inorganic sulfur has been removed.

Powell's work on sulfur forms in coal did not include North Dakota lignite. The Bureau of Mines Technical Paper 695 gives the sulfur distribution for Velva Lignite; and William McMillan, Chemist with the North Dakota Research Foundation, analyzed three lignite samples from the Garrison Damsite for pyritic and sulfate sulfur, organic sulfur, and total sulfur. These analyses and those which I have made are found in the accompanying table.

From these analyses it is seen that North Dakota lignite has a relatively low amount of total sulfur. Reviewing circulars of the University School of Mines, it is found that the average sulfur content of 546 mines was 0.65%. The sulfate sulfur was found to be very low and the pyritic content was a small percentage of the total. The organic sulfur in most of the analyses was more than 50% of the total content. The assumption that one-half of the sulfur in coal is organic is approximately correct for most coals. There are advantages in having the sulfur in organic combinations rather than as pyrites. It is the iron pyrite that influences clinker formation, and that aids spontaneous combustion. Another advantage of having the great part of the total sulfur in the form of organic compounds is that the sulfur content for a certain mine or area is more uniform than it would be if pyrites were present.

Further evidence that the sulfur in North Dakota lignite is mostly in the organic form is shown by analyses of samples of North Dakota lignite reported in the Bureau of Mines Technical Paper 700. From these analyses it is found that as the size of the coal is decreased by crushing there is an increase in the ash content, but the quantity of sulfur changes little, and is therefore independent of the ash. For example:

Wilton Lignite	Size	Ash, %	Sulfur, %
	8 " lump	4.7	.6
	4 " by 8 "	5.0	.5
	2 " by 3 "	5.8	.6
	½" by 1½"	5.9	.6
	2 " slack	7.0	.6
<b>Quality Lignite</b>			
	4 " lump	4.1	.2
	2½" by 4 "	4.5	.2
	1¾" by 2½"	6.3	.2
	¼" by ¾"	5.5	.2
	¼" slack	5.9	.2

Little is known about the composition of organic sulfur compounds in lignite or any other rank of coal. And there is no evidence of a definite relationship in the occurrence of organic and pyritic sulfur.

From this study it seems that sulfur is an undesirable element in coal, and it is classed with impurities. And although it may not be responsible for all the troubles attributed to it, it contributes nothing to making a coal a better fuel. Therefore, the low amount of sulfur in North Dakota lignite is of no small advantage to coal users.

### SULPHUR IN NORTH DAKOTA LIGNITE

Percentage of Forms of Sulfur (As received basis).

Mine	Sulfate			Organic	Total
	Sulfate	Pyritic	Pyritic		
1. Velva .....	0.019		0.0015	0.156	0.20
2. Garrison Damsite		0.30		0.70	1.00
3. Garrison Damsite		0.10		0.40	0.50
4. Garrison Damsite		0.10		0.40	0.50
5. Garrison .....	0.01		0.02	0.30	0.33
6. Velva .....	0.02		0.15	0.17	0.34
7. Baukol-Noonan .....	0.02		0.06	0.27	0.35
8. Dried Kincaid .....	0.01		0.10	0.35	0.46
9. Kincaid .....	0.01		0.04	0.28	0.33
10. Lehigh .....	0.07		0.47	0.34	0.88
11. Velva .....	0.01		0.07	0.14	0.22

Sample 1—analyzed by Bureau of Mines, Technical Paper 695 (1946).

Samples 2, 3, 4—analyzed by William McMillan, North Dakota Research Foundation.

Samples 5, 6, 7, 8, 9, 10, 11—analyzed by Adelynn Magnusson, UND.

### “HUMIC ACIDS” FROM WEATHERED LIGNITE (LEONARDITE)

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At the 12th Annual meeting of this Academy of Science, held at the Agricultural College, in 1920, the writer presented a brief report under the title, “Lignite Jelly—A Natural Reversible Colloid.” The material described had been sent to him in the form of a black gel, found in a roadside ditch near lignite deposits in western North Dakota.

When allowed to dry, the material formed hard, lustrous lumps resembling coal. When placed in water, these lumps again dispersed, forming a dark brown to black solution. The product was correctly diagnosed as material formed from weathered lignite by the action of alkaline water.

It was brought to the attention of the late Professor A. G. Leonard, then head of the Geology Department of the University of North Dakota and State Geologist, with the suggestion that many tons of the material would be found on the surface of lignite beds exposed to prolonged weathering.

Professor Leonard made a careful study of these surface layers, which are commonly discarded by miners as inferior coal. In recognition of his work, the mineral is now designated by geologists as "Leonardite."

Soon after, Professor Dove, of the Geology Department, developed the material commercially as a wood stain, which was manufactured and sold under the trade name of "Dakolite." Similar stains have been produced in Germany from brown coals.

This report attempts to summarize the information gained from investigations of the material extending over a considerable period of time. In 1932, with the capable assistance of Mr. Cecil O. Lohn, then a graduate assistant in Chemistry, the composition was determined by careful analysis, and investigations were made of the colloidal properties and behavior.

The material was purified by leaching the crude product with water rendered alkaline with sodium hydroxide. After prolonged sedimentation, the dark liquid was decanted from the insoluble residue, and treated with sufficient hydrochloric acid to cause flocculation and precipitation. After standing for several days, the precipitate was filtered by suction and thoroughly washed, first with successive portions of 6 normal hydrochloric acid and then with successive portions of distilled water until the washings were finally chloride-free. It is difficult to free the gelatinous precipitate from adsorbed ions, but it can be done by sufficiently prolonged washing. The washed precipitate was allowed to dry for 24 hours at room temperature, after which it was dried in an electric oven at 105 degrees for three hours. The purified product was then preserved in a desiccator.

#### Analysis of the Product

Five representative samples, independently prepared, gave the following results. Oxygen values were determined by differences.

#### Summary of Average Percentage Composition<sup>1</sup>

Constituent	Per cent	Atomic Weight	Atomic Ratio
Carbon .....	61.949	12.000	5.162
Hydrogen	2.824	1.008	2.801
Nitrogen .....	1.589	14.008	0.1134
Oxygen .....	29.582	16.000	1.849
Ash	4.056	—————	—————

#### Calculated Empirical Formula

While such a formula has little significance, an approximate formula is obtained by multiplying each atomic ratio by 10. This gives  $C_{51}H_{28}O_{18}N$ , assuming that the material is a single substance—which is highly improbable.

The high carbon to hydrogen ratio 51/28, indicates that the molecules cannot be saturated aliphatic chains. Even if they were un-

<sup>1</sup> Cecil O. Lohn, Unpublished Master's Thesis, University of N. Dak., (1932).

saturated chains, the unsaturation would be extreme, which is not observed in the behavior of the material. They must, therefore, consist of cyclic compounds of high molecular weight, as also indicated by their colloidal behavior.

The acid character of these "humic acids" is shown by their behavior with bases, and their precipitation from alkaline solutions by strong acids. The high oxygen content suggests the probability that they contain several carboxy groups, indicating that they are weak polybasic acids. Oxygen may also occur in hydroxyl groups, and possibly even as a link in ring structures.

The solutions of the alkaline salts of these humic acids are largely hydrolyzed, and the insoluble acids set free by the hydrolysis are dispersed in colloidal form by the strong peptizing action of the humic salts, as well as that of the free base. Hydrolysis with dilute acids failed to indicate any simple protein cleavage products, or monosaccharides from carbohydrate structures.

#### **Colloidal Behavior of the "Humic Acids"**

Colloidal sols were prepared by peptizing the insoluble acid residue with the minimum amounts of sodium hydroxide required, after which the solutions were subjected to prolonged dialysis in prepared collodion bags. In a series of determinations with weighed samples of the dried colloid, using successive small volumes of 0.1012 normal sodium hydroxide, it was found that the number of milligrams of sodium hydroxide required by 1 gram of the colloid varied between 155.6 mgs and 215.6 mgs for different samples. These variations were considered to indicate differences in the degree of dispersion of the colloid, due to varying conditions under which the acid precipitations had been made, rather than to corresponding differences in chemical composition.

Some of the samples were dialyzed for a month or longer, with frequent changes of distilled water. The sols obtained exhibited typical colloidal behavior. They show the Tyndall effect with light, and the Brownian movement when viewed with the ultramicroscope.

Experiments on electrical migration indicated that the colloidal micelles carry negative electric charges. They were precipitated by positive ions, in general following the valence rule; but they are less sensitive to these ions than are most typical suspensoids, showing some similarity to the behavior of emulsoids, possibly due to the presence in them of such strongly lyophilic groups as hydroxyl and carboxyl. Addition of relatively large amounts of strong acids caused a reversal of the charge on the particles, as shown by the change in direction during electrical migration. From electrometric titration curves, the isoelectric points were estimated to lie between pH 2.6 and pH 2.9. Electrometric measurements of the pH with hydrogen-calomel electrodes on 2 independently prepared sols gave values of 5.87 and 6.55 respectively. Since water suspensions of precipitated humic acids show a pH of about 4, the values obtained with the sols would



seem to indicate that the hydrolysis of the sodium humates was not complete even after very prolonged dialysis. It may also explain the difference in pH observed with different sols.

The composition of these lignite humic acids shows a striking similarity to that of oxidation products produced from bitumenous coals. In a private communication, Dr. Henry C. Howard, of the Carnegie Institute, at Pittsburgh, stated, "The ultimate composition of the product prepared from lignite jelly, under your direction, is very similar to our oxidation products from bitumenous coal." A comparison of the two products is shown in the following table:

<b>Percentage Composition</b>		
	<b>Humic Acids from Lignite</b>	<b>Oxidation Products from Bitumenous Coal</b>
Carbon .....	61.95	60.75 - 62.17
Hydrogen .....	2.82	3.12 - 3.31
Nitrogen .....	1.59	2.72 - 4.05
Oxygen .....	29.58	28.9 - 31.9
Ash .....	4.05	0.95 - 1.44

The higher values of nitrogen in the coal products are due to the dilute nitric acid used as oxidizing agent, a part of which is always fixed in the products. Lower ash values, likewise may result from the action of the acid.

The chief interest in these humic acids is due to their possible role in the natural process of coal formation. They are believed to be products formed in the first stage of a series of reactions, resulting in the final conversion of vegetable matter into coal. Many have tried to show that they are derived from lignin; but the consensus of opinion now seems to be that they are formed from cellulose itself.

Notwithstanding the vast amount of work done on them, these humic acids comprise a group of substances about which, even yet, comparatively little is known with certainty; but it is hoped that further investigation of them may finally give the answers to many important questions concerning the mechanisms of coal formation from vegetation.

## THE ACETYLATION OF MONOCARBOXYLIC ACIDS WITH KETENE

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The reaction of ketene with both aliphatic and aromatic acids in the preparation of mixed anhydrides was first reported by Hurd and Dull<sup>1</sup> in 1932:  $\text{CH}_2=\text{C}=\text{O} + \text{R}-\text{COOH} = \text{R}-\text{CO}-\text{O}-\text{CO}-\text{CH}_3$ . Since this reaction usually occurs at room temperature, in the pres-

<sup>1</sup> Hurd, C. D., and Dull, W. P., *J. Am. Chem. Soc.*, 54, 3427 (1932).

ence of inert solvents, without the aid of catalysts it afforded a convenient means of preparing and studying other mixed anhydrides. Likewise, since inert solvents were permissible it afforded a practical method of acetylating even solid organic acids. The ketene used was generated by the pyrolysis of acetone using the lamp previously described by Dunbar and Bolstad<sup>2</sup>. Thirty organic aliphatic and aromatic acids were each individually placed in the gas adsorption apparatus designed by Dunbar and Bolstad<sup>3</sup> and treated until a theoretical excess of ketene had been added. Dry ether was used as a solvent for the solid acids. Eight such mixed anhydrides, previously prepared with ketene, were again produced and former constants checked or new constants recorded. Twenty-two additional mixed anhydrides were prepared and their physical constants recorded. Of this latter group, nineteen have never been previously reported in the literature and the remaining three have never been previously produced by the use of ketene. These compounds and constants are recorded in Table I.

The mixed acid anhydrides were purified by vacuum distillation or recrystallization from petroleum ether. Many of the constants of the known anhydrides do not agree with those previously reported. When the size of the two alkyl or aryl groups were decidedly different, the mixed anhydrides showed a decided tendency to disproportionate into the two corresponding simple anhydrides:  $2 R-CO-O-CO-R' = R-CO-O-CO-R + R'-CO-O-CO-R'$ . This behavior greatly complicated the purification and determination of physical constants, and frequently required the immediate use of these anhydrides after preparation. Fortunately, Whitford<sup>4</sup> has devised a satisfactory method for evaluating the purity of a mixed anhydride, and this test when applied to the freshly prepared anhydrides gave satisfactory results. Oxalic acid and pyridine, saturated with carbon monoxide and carbon dioxide, when treated with a mixed acid anhydride reacts as follows:  $(COOH)_2 + R-CO-O-CO-CH_3 + (pyridine) = CO + CO_2 + CH_3COOH + RCOOH$ . The gases liberated constitutes a measure of the purity and requires only about fifteen minutes for completion.

Aniline has been found to react with freshly prepared mixed anhydrides to form the anilide of the acid having the least electron attraction within its radical (1):  $CH_3-CO-O-CO-C_6H_5 + C_6H_5-NH_2 = C_6H_5COOH + CH_3-CO-NH-C_6H_5$ . Aryl radicals have a greater electron attraction within the molecule than the alkyl radical. Also the methyl group has a slightly greater electron attraction than the propyl group. When many of these mixed anhydrides were allowed to stand for twenty-four hours or more, prior to reacting with aniline, both anilides resulted, indicating that the anhydride must have undergone a molecular disproportionation. Pure mixed an-

<sup>2</sup> Dunbar, R. E., and Bolstad, L. L., *J. Org. Chem.*, **9**, 219 (1944).

<sup>3</sup> Dunbar, R. E., and Bolstad, A. N., *Anal. Chem.*, **18**, 337 (1946).

<sup>4</sup> Whitford, E. L., *J. Am. Chem. Soc.*, **47**, 2934 (1925).

TABLE I. Mixed Acetic (Alkyl-Aryl) Anhydrides.

Alkyl-Aryl Group	B. P. - M. P.	<sup>20</sup> n <sub>D</sub>	<sup>20</sup> D <sub>4</sub>	Mr <sub>D</sub>	M. P. Anilide
Propionic <sub>1</sub>	31-2 <sub>5</sub>	1.3963	1.013	27.43	78-82°
Butyric <sub>1</sub>	41-3 <sub>5</sub>	1.405	1.00	31.87	87-93°
iso-Butyric <sub>1</sub>	35.7-7 <sub>5</sub>	1.400	0.994	31.70	112-15°
Valeric <sub>1</sub>	55-7 <sub>6</sub>	1.4100	0.988	36.28	95-100°
iso-Valeric <sub>1</sub>	48-60 <sub>6</sub>	1.4053	0.982	35.99	98-110°
Caproic <sub>1</sub>	56-72 <sub>10</sub>	1.401	0.955	41.22	100-5°
Benzoic <sub>1</sub>	————	1.4955	1.501	43.37	100-10°
Stearic <sub>1</sub>	54+	1.427 <sub>70</sub>	0.885 <sub>70</sub>	96.93	————
Formic <sub>2</sub>	27-8 <sub>10</sub>	1.388	1.120	18.26	112.0°
Enanthic <sub>2</sub>	————	1.414	0.943	45.62	55-83°
n-Caprylic <sub>3</sub>	————	1.421	0.940	50.16	45-65°
Pelargonic <sub>3</sub>	————	1.422	0.935	54.33	————
n-Capric <sub>3</sub>	————	1.428	0.932	59.23	63-8°
Lauric <sub>3</sub>	————	1.433	0.914	68.84	78-83°
Myristic <sub>3</sub>	46-7 +	1.432 <sub>35</sub>	0.899 <sub>35</sub>	78.38	53-7°
Palmitic <sub>2</sub>	49-51+	1.432 <sub>90</sub>	0.869 <sub>60</sub>	89.08	60-3°
Crotonic <sub>3</sub>	————	1.438	1.003	33.53	107-8°
Undecyclonic <sub>3</sub>	————	1.436	0.933 <sub>27</sub>	63.38	108°
Phenylacetic <sub>3</sub>	————	1.496 <sub>23</sub>	1.090 <sub>25</sub>	47.28	92-6°
Diphenylacetic <sub>3</sub>	————	1.524	1.10	70.79	————
Cyclohexylacetic <sub>3</sub>	————	1.444 <sub>26</sub>	1.019	48.02	92-6°
Cyclohexylpropionic <sub>3</sub>	————	1.449 <sub>22</sub>	1.01 <sub>22</sub>	52.67	83-5°
Cyclohexylbutyric <sub>3</sub>	————	1.450 <sub>22</sub>	0.994 <sub>22</sub>	57.44	70-110°
Cyclohexylcaproic <sub>3</sub>	————	1.454	0.976	66.67	90-3°
Cinnamic <sub>3</sub>	120-5+	————	————	————	93-103°
alpha-Naphthoic <sub>2</sub>	————	1.568	1.151	60.92	————
o-Toluic <sub>3</sub>	————	1.513	1.125	47.57	99-103°
m-Toluic <sub>3</sub>	————	1.504	1.102	47.82	105-6°
p-Toluic <sub>3</sub>	————	1.516	1.118	48.14	165-70°
2, 4-Dichlorophenoxyacetic <sub>3</sub>	————	1.513	1.308	60.44	93.5°

<sub>1</sub> Previously prepared with ketene but limited properties reported.

<sub>2</sub> Previously prepared but not by the use of ketene.

<sub>3</sub> New additional mixed anhydrides.

+ Melting points, C.

hydrides show little reaction with sodium carbonate solution,<sup>5</sup> while an equal molecular mixture of two different anhydrides react readily. The three tests listed immediately above, as well as the melting points of the anilids, confirms our conclusion that the freshly prepared mixed anhydrides were relatively pure and were not mixtures of two distinctly different anhydrides. While combustion analyses could not be conclusive, it is encouraging to note that these results, as well as molecular refractions, substantiate the above conclusions.

<sup>5</sup> Behal, A., J. Chem. Soc., 76, 8 (1900).

The resulting mixed anhydrides, besides having considerable theoretical significance, are useful in the Friedel-Crafts reaction, for the preparation of mixed esters of polyhydroxy compounds, and by disproportionation for the synthesis of high molecular weight simple anhydrides, together with acetic anhydride as a by-product.

## SYNTHETIC PLANT GROWTH REGULATORS AND FLOUR PROTEINASE ACTIVITY

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The proteolytic enzymes of flour have been investigated for many years but their influence on gluten during fermentation is still not too clear. The weight of evidence is in favor of these enzymes playing some part, but not necessarily the major role, in the phenomenon of dough ripening. Two naturally occurring reducing substances, glutathione and cysteine, greatly accelerate proteolysis, probably because the proteolytic enzymes of flour are present in a latent form. Oxidation of these promoters, or inhibition of proteolytic enzymes, would cause a reduction in proteolysis.

The increasing importance of plant growth regulators including weed killers has stimulated investigations regarding the effects of these substances upon the physiological and chemical properties of many types of plants. This investigation offers evidence of inhibition of proteolytic activity by synthetic plant growth stimulants. Concentrations of 150 p. p. m. of the stimulants in gluten dispersions in acetic acid resulted in slight inhibition of proteinase activity. Complete inactivation was attained by removing the gluten from dispersion with sodium alpha-naphthyl acetate, and redispersion in 0.1 N acetic acid. Partial reactivation was secured by two successive reprecipitations with potassium sulfate, followed by redispersion in acid. Inactivation was also secured by dispersion in 12% sodium alpha-naphthyl acetate, as in sodium salicylate.

The solubility in 0.1 N acetic acid of glens prepared from flour-water doughs incubated at 30° C. was reduced by concentrations of 0.09% sodium phenyl acetate, indicating flour proteinase inactivation. At higher concentrations the solubility was lowered probably because of protein coagulation. To remove the effect of proteolysis, the doughs with increasing concentrations of stimulant were incubated for 24 hours at 0° C. Sodium phenyl acetate concentrations of 2.0% and 3.0% caused a decrease in gluten solubility indicating the point where coagulation commences.

Mixograms were secured on the flour-water doughs to obtain information on changes in their physical properties during mixing. The results of these curve patterns apparently showed proteinase inhibition by the stimulants, but the changes in protein solubility yielded more reliable data.

## A METHOD OF AUTOMATIC DEHYDRATION FOR HISTOLOGICAL TECHNIQUE<sup>1</sup>

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*University of North Dakota School of Medicine*

Heretofore the mechanics commonly involved in the process of dehydration have consisted of manually or mechanically transferring tissues through a series of dehydrating chambers containing the dehydrating agents, or of leaving the tissues in a single chamber and periodically changing the surrounding dehydrating fluids.

A new method is now introduced whereby the tissues are continuously and automatically bathed in a continuous flow of anhydrous dehydrating agent. This is accomplished by the use of an extraction apparatus equipped with a Soxhlet Extractor having an overflow drainage rather than the usual siphon type drainage. The tissues are placed in the modified Soxhlet Extractor, which is then filled with dehydrating fluid. A funnel placed in the extractor is used to carry the condensate to the bottom of the chamber to insure a continuous change in fluid throughout the depth of the extractor. The flask is filled approximately three-fourths full of dehydrating agent, and 80-100 g of calcium carbide (depending upon the moisture content of the dehydrating agent and tissues) is also added to insure a completely anhydrous condensate. A thermostatically controlled hot plate with a variable temperature control is used as the source of heat. Thus, as the dehydrant is refluxed, the condensate (anhydrous dehydrating agent) enters the bottom of the extracting chamber, circulates through the tissues and finally spills out through the overflow drainage back down into the flask. The apparatus should be operated under a hood, or else a rubber hose should be attached to the top of the condenser to carry off the fumes.

This method offers several definite advantages. Dehydration is more complete. A flow of circulating fresh anhydrous dehydrant completely surrounds the tissues at all times. This is an important consideration, since incomplete dehydration is a frequent cause of shrinkage and hardening of tissues within paraffin blocks after they have been cut off and put away. If a gradually increasing concentration of the dehydrant is desired, one need only fill the extraction chamber with the desired starting concentration of dehydrant at the beginning of the procedure. This dehydrant then will be very gradually replaced by completely anhydrous dehydrating agent, giving the effect of transferring the tissues through a finely graded series of reagents.

It is automatic. A manual transfer of the tissues is not necessary, nor is a manual change of dehydrating fluid. The tissues may be placed in the apparatus towards evening and left unattended until the following morning, when they will be dehydrated and ready for further processing.

<sup>1</sup> This work was supported in part by a National Institutes of Health grant.

It is inexpensive. Only small amounts of dehydrating agent are necessary, since the dehydrant is being continually refreshed as the process proceeds. The apparatus itself is of simple design, and any glass blower can make it at a nominal cost simply by altering a regular Soxhlet Extractor as indicated.

Although most of the commonly used dehydrating agents are adaptable to this procedure, it is preferable to use those having relatively low boiling points, so that the tissues may be safeguarded against any heat damage. We have had good success with acetone with the above method and at present are testing various other reagents, particularly those which can be used both for the dehydration and the clearing process, thus extending the automaticity of the procedure.

## THE CULTURAL DIAGNOSIS OF MYCOBACTERIUM TUBERCULOSIS

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The demonstration of tubercle bacilli in the sputum or body fluids of a patient is the surest method of diagnosing tuberculosis.

The three laboratory methods most widely used are (1) direct microscopic examination of smears made from specimens, (2) cultural methods using solid media and, (3) animal inoculation.

Microscopic examination is rapid, simple and inexpensive; however, it is not sensitive enough when only a few tubercle bacilli are present in a specimen; also, it is impossible to say with certainty that acid-fast organisms observed are really tubercle bacilli since it is practically impossible to differentiate non-pathogenic acid-fast organisms from tubercle bacilli.

Cultural technique, using the Jensen-Holm modification of Lowenstein's medium, offers an efficient routine diagnostic method. The trained bacteriologist can differentiate with a fair degree of certainty non-pathogenic from pathogenic acid-fast bacilli.

The morphological appearance of the various strains of tubercle bacilli are as listed below.

Type	Optimum Temperature	Appearance of Growth	Characteristics of Growth	Color
Human	37°C	3 weeks	Eugonic - rough Large colonies Dry - friable Cabbage appearance	Buff
Bovine	37°C	4 weeks	Dysgonic - smooth Pyramidal colonies	Pale-White
Avian	40°C	2 weeks	Eugonic - smooth Hemispherical colonies	Yellow (faintly)
Saprophytes	20-40°C	1 week	Eugonic - smooth and/or rough Soft, creamy colonies	Usually chromogenic

These characteristics will hold true for first isolation (not on stock cultures.)

Animal inoculation is a valuable method of diagnosing tuberculosis, not only as a procedure for typing of strains but also to rule out definitely acid-fast saprophytes. All body fluids are subjected to animal inoculation as well as cultural techniques.

Preliminary results of cultural work indicate that the cultural method on digested specimens is far superior to the microscopic examination of sputa specimens and more efficient in diagnosing gastric washings.

**TABLE I.**

**Comparison Microscopic Examination and Cultural Method on Sputa Specimens for Tubercle Bacilli.**

Result	No. Specimens	% of Total
Cult. + ; mic. +	20	5.9
Cult. - ; mic. -	250	73.6
Cult. + ; mic. -	32	9.5
Cult. - ; mic. + .....	0	—
Cult. contam.; mic. -	36	10.5
Cult. contam.; mic. +	2	.5
Total .....	340	100.0

**TABLE II.**

**Comparison Microscopic Examination and Animal Inoc. on Body Fluid Specimens for Tubercle Bacilli.**

Result	No. Specimens	% of Total
Cult. - ; animal -	150	72.6
Cult. + ; animal +	14	6.8
Cult. - ; animal +	3	1.4
Cult. + ; animal - .....	15*	7.3
Cult. contam.; animal +	3	1.4
Cult. contam.; animal -	16	7.7
Cult. - ; animal died .....	6	2.8
Total .....	207	100.0

\* All gastric washings (11.8%).

**TABLE III**

**Results of Cultures All Specimens**

Pos.	%	Neg.	%	Uns.	%	Total
81	14.8	409	74.8	57	10.4	547

**Results of Microscopic Examination**

Pos.	%	Neg.	%	Uns.	%	Total
28	6.6	392	93.4	—	—	420

**Results of Animal Inoculation**

Pos.	%	Neg.	%	Uns.	%	Total
19	9.2	181	87.8	6	3.0	206

The cultivation method offers a more sensitive technique for determining the presence of small numbers of tubercle bacilli in pathological material.

On the Lowenstein-Jensen medium, the trained bacteriologist can differentiate with a fair degree of reliability pathogenic from non-pathogenic acid-fast bacilli.

In our hands the cultural method thus far has proven to be a much more reliable method of making a positive diagnosis of tuberculosis, as shown in the above tables. A total of 47 positives were obtained by the cultural technique which were found to be negative by other laboratory methods and would have been reported as negative prior to the routine use of cultural methods.

## THE PRECIPITATION OF AMINO ACIDS FROM AQUEOUS SOLUTION BY USE OF DIBENZOFURAN-2-SULFONIC ACID

*Ray T. Wendland and Charles H. Smith*

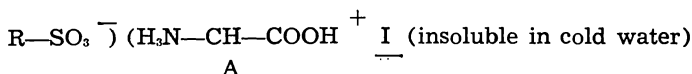
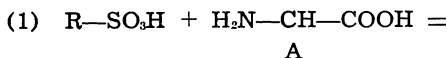
*School of Chemical Technology, North Dakota State College*

The work reported here concerns the preliminary investigation of dibenzofuran-2-sulfonic acid as a reagent for the precipitation of amino acids<sup>1</sup> It has been found here that this compound is a general precipitant for cations, and hence its possible utility in the recovery of amino acids from aqueous solutions has been suspected.

The present report is drawn largely from the experimental findings of Charles H. Smith, who has been studying the reactions of pure amino acids, and John Rode (M.S. North Dakota State College, December, 1948) who prepared dibenzofuran sulfonic acid salts of many of the common amines.<sup>2</sup>

The basic problem under investigation is the recovery of amino acids from protein hydrolyzates. For this purpose it is necessary first to gather quantitative data on the solubilities of the sulfonate derivatives of the amino acids, and this part of the project may be considered practically completed.

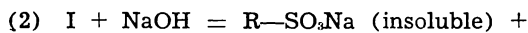
The reactions which we are attempting to carry out are the following:



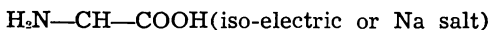
<sup>1</sup> Wendland, R., Smith, C. H., and Muraca, R., *J. Am. Chem. Soc.*, **71**, 1593 (1949).

<sup>2</sup> Rode, J. W., *A Study of Dibenzofuran-2-Sulfonic Acid Derivatives of Amines*, M. S. Thesis, North Dakota State College (December, 1948).





(hot solution)



A

In equations (1) and (2) R = dibenzofuryl, A = amino acid residue.

Our work shows that most of the salts of the type of I are very slightly soluble in cold water but can be dissolved in hot water. Furthermore, we know that the reaction (2) has reasonable chance of success because of the very low solubility of sodium dibenzofuran sulfonate, and the fact that amino acids can be kept in solution at a pH slightly above 7.0, or for those which are stable to alkali the pH may be raised even more to assure solubility of the sodium salts.

#### Procedure for Preparation of Amino Acid Salts of Dibenzofuran-2-Sulfonic Acid

A weighed amount of the amino acid was dissolved in a minimum amount of water. In the case of the slightly soluble amino acids, one equivalent of hydrochloric acid was added to effect solution. In either case an equivalent amount of a saturated solution of dibenzofuran sulfonic acid was added (normality = 0.30 to 0.33). The salt which precipitated at room temperature was collected, re-crystallized, and prepared for analysis. (For several of the amino acids the formation of crystals was slow, but in no cases did formation of oils occur.)

In the few cases where precipitation of the amino sulfonate did not occur at room temperature the mixture was chilled at zero degrees C. to induce crystallization. Two of the amino acids (1-proline and 1-tyrosine) failed to give any precipitation at this point, while a third (glutamic acid) yielded a precipitate but this appeared to be mostly the original sulfonic acid. Its analysis for sulfur was always high (11-12 percent), whereas, that required for the glutamic acid dibenzofuran sulfonate is 8.1 percent, that for the free sulfonic acid is 12.9 percent. Furthermore, microscopic examination of this precipitate showed the presence of two crystalline forms, the more abundant one looking much like the free sulfonic acid. No explanation is given here for this anomalous result.

The method adopted for determining solubilities was that of removing aliquot portions of the supernatant liquid from a saturated solution ( $t = 0^\circ\text{C}.$ ) of the amino acid sulfonate. These aliquots were evaporated to dryness in tared bottles by standing in a vacuum desiccator over sulfuric acid. In this way the residual solid remaining in the saturated solution could be very easily determined with a degree of precision sufficient for our purpose.

The salts prepared were analyzed for sulfur by the Parr bomb combustion technique, and for nitrogen by the micro Dumas method. The quantitative data on the analysis and solubility of the salts are summarized below.

### Conclusion

It has been shown possible in this study to precipitate most of the amino acids from dilute aqueous or acid solutions by the use of a new reagent, dibenzofuran-2-sulfonic acid. We have developed in this laboratory a very simple method of preparation of this valuable compound so that it may now be considered a readily available reagent of low cost.

### DIBENZOFURAN SULFONIC ACID SALTS OF AMINO ACID

No.	Compound (Crystal Habit)	Solubility g./100 g. H <sub>2</sub> O		Analysis, %			
		M. p. ° C.	at 0° C.	Sulfur Calcd.	Sulfur Found	Nitrogen Calcd.	Nitrogen Found
1.	d1-Alanine DBF-2-S Needle clusters	247-8	2.3	9.30	9.16	4.15	4.41
2.	1-Arginine (DBF-2-S) <sub>2</sub> Needle clusters	d.232-6	1.3	9.55	9.38	8.36	8.27
3.	Asparagin DBF-2-S Hexagonal plates	d.273-5	3.1	8.43	8.21	7.37	7.07
4.	Aspartic acid DBF-2-S Hexagonal plates	214	2.0	8.41	8.80	3.67	3.36
5.	Creatinine DBF-2-S Rectangular plates	264d.	0.85	8.87	8.62	11.62	11.83
6.	1-Cysteine DBF-2-S Irregular plates	218d.	4.0	17.36	16.89	3.79	3.31
7.	1-Cystine (DBF-2-S) <sub>2</sub> Amorphous	d.207	1.0	17.41	17.13	3.80	3.87
8.	d-Glutamic acid DBF-2-S	salt will not precipitate from solution					
9.	Glycine DBF-2-S Rods	d.234-5	8.0	9.92	10.17	4.33	4.33
10.	1-Histidine (DBF-2-S) <sub>2</sub> Needles	240-1	2.5	9.85	9.44	6.45	6.13
11.	1-Hydroxyproline DBF-2-S Rectangular plates	225	4.75	8.44	8.11	3.7	4.0
12.	iso-Leucine DBF-2-S Plates	197-8	0.8	8.45	8.06	3.69	3.80
13.	1-Leucine DBF-2-S Rods	220	0.5	8.50	8.25	3.69	3.94
14.	d1-Lysine (DBF-2-S) <sub>2</sub> Irregular plates	228-30	1.0	9.98	9.64	4.36	4.41
16.	Nicotinic acid DBF-2-S Rectangular plates	194	0.5	16.13	16.17	3.52	3.62
16.	Nicotonic acid DBF-2-S Plates	223	6.4	8.62	8.23	5.16	5.44
17.	d1-Phenylalanine DBF-2-S Needle Clusters	228d.	0.25	7.76	7.81	3.39	3.14
18.	1-Proline DBF-2-S	salt will not precipitate from solution					
19.	Serine DBF-2-S Prisms	207-10d.	3.5	9.05	9.22	3.95	3.81
20.	d1-Threonine DBF-2-S Rectangular Plates	200	3.2	8.73	8.65	3.81	3.76
21.	1-Tryptophane DBF-2-S Needles	234d.	0.4	7.09	7.01	6.19	6.19
22.	1-Tyrosine DBF-2-S	salt will not precipitate from solution					
23.	d1-Valine DBF-2-S Rods	196	3.9	8.78	8.51	3.83	3.99

## MICROBIOLOGICAL DETERMINATION OF AMINO ACIDS

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It has not been many years since the determination of amino acids in proteins was a complicated and time consuming process. First the proteins in plant material had to be separated from other substances such as starch, then the protein was hydrolyzed by either an acid or a base, and finally the amino acid was determined by chemical means. This procedure often required very large samples in making an assay of all the amino acids.

In latter years the microbiological method, which was first used in the determination of the B complex vitamins, has been applied to the determination of amino acids in proteins. With this method a very small sample is required to make a complete assay of the amino acids. The sample can be hydrolyzed directly without removal of the starch, and the hydrolysate used for amino acid analyses.

Some organisms require certain amino acids for growth. The organism that has the highest requirement for a particular amino acid is used in the assay. The organism most commonly used for this assay belonged to the *Lactobacteriaceae* Family. All of these organisms used produce acid upon growth. If the media used is adequate in all respects with the exception of the amino acid being assayed, the growth of the organism which is determined by the amount of acid produced is a direct function of the limiting amino acid. With the use of a particular amino acid-free but otherwise complete basal medium, growth responses are compared quantitatively in standard and unknown solutions.

### Procedure

In this laboratory the determination of methionine has been used more extensively than any other, although the determinations for the other amino acids are very similar. The organism used is *Streptococcus Faecalis*.

The medium must contain all the necessary growth factors with the exception of methionine. The following constituents are used in the medium: Glucose, which is a source of energy; sodium citrate and acetate, which serve as buffers; salts such as the salts of potassium, iron and phosphate; purine and pyrimidine bases, vitamins belonging to the B complex group; amino acids with the exception of methionine; and water.

This medium is adjusted to a pH 7 and 1 ml. is added to tubes containing known amounts of methionine and to tubes containing known amounts of the hydrolyzed protein extract. Each tube is made up to a total volume of 2 ml. These tubes are capped and sterilized at

15 lbs. pressure for ten minutes, cooled, and then inoculated. The organism which is used for inoculation is grown on enriched medium for 18 hours, centrifuged down, the medium poured off, dispersed in a physiological saline solution, and then used for inoculation.

After inoculation the tubes are incubated for 72 hours at 37 C. in a water bath. At the end of the period of incubation the amount of acid produced is determined by titration with .05 N. alkali. To obtain a standard growth curve the amount of alkali used to neutralize the acid produced versus the amount of methionine present is plotted on a graph. Then the amount of alkali used to neutralize the the amount of acid produced in the unknowns is compared to the alkali used in the standard curve and thus the methionine content is obtained for the unknown.

It is evident from the above procedure that an assay of this type requires a large amount of pipetting and titration. This part of the assay would be rather laborious and tiresome, however, recently there was put on the market an instrument, which is not very largely advertised, known as the Cannon Automatic Dispenser and Titrator. Both dispensing and titration are speeded up through the use of this instrument.

This device operates on the principle of analogy. Like all analogue mechanisms one thing is equated to another. In this instance the things equated are time and volume. An interval of time is used to measure a quantity of liquid, since the volume of fluid which will flow through a given orifice will depend upon the length of time which it is allowed to flow if the temperature, viscosity and pressure of the liquid remain constant.

Temperature and viscosity are not ordinarily factors which require control since measurement is usually carried out with solutions which are at room temperature. The viscosity is inherent in the liquid being measured and must be allowed for in the initial calibration for any given liquid.

The size of orifice, the pressure and the duration of flow are factors for which control is provided for in this device and which may be varied at will to give any desired volume. Changing the size of the orifice provides for major changes in the volume delivered. This is a discontinuous method for effecting changes in the volume delivered useful for adapting the machine to widely different uses. Variations in pressure will also control the volume delivered in a given time interval and provide a fine, continuously variable means of adjusting the volume delivered. The time intervals are selected for convenience and speed in operation and the orifice chosen and the pressure adjusted to give the volume per interval desired.

The pressure is regulated by a pressure regulator which works on the following principle: The mercury of a mercury monometer completes an electric circuit whenever the predetermined pressure is reached. This operates a small valve to vent air until the pressure

has dropped sufficiently for the mercury to fall a small amount and break the contact. To lessen the fluctuations in pressure to which the liquid being dispensed is exposed a check valve is placed in the system. This check valve, while passing air to the fluid reservoir, does not allow an outrush of air every time the venting valve is opened.

The timer controls the third of the variables that has been mentioned. The intervals during which current flows through solenoid and during which liquid flows through the orifice are represented by segments cut from a paper disc which allows a small spring to contact a metal plate. Plate and disc are rotated at constant speed by a synchronous motor.

With reasonable care in the adjustment of the controls an accuracy of about 1% may be achieved. That is, in a series of volumes delivered from a single segment none will differ more than 1%.

This apparatus may also be used to measure the amount of a reagent solution required to titrate a sample. The advantages over titration with a burette are increased speed, lessened fatigue, and greater accuracy.

In addition to the equipment used for dispensing a magnetic counter is required. This counter is actuated by impulses generated by a punched paper disc which replaces the notched paper disc used for dispensing. The solenoid valve plug and the counter plug are rearranged so that when the foot-switch is closed the valve opens and remains open until the foot-switch is released. The length of time the valve is open and consequently the amount of reagent delivered is recorded by the counter.

Instead of measuring the volume of reagent delivered by a certain number of counts it is simpler to titrate standards to the desired end point and use the counts thus obtained in calculating the unknowns. In microbiological assay work the standard curve is titrated and the results plotted in terms of the arbitrary numbers recorded by the counter. The values of the unknown samples may then be read from this curve in the same manner as if the titrations had been recorded in milliliters of alkali.

The end point may be determined by any of the conventional methods, but in this laboratory, a quinhydrone-platinum electrode-calomel half cell system connected to an inexpensive galvanometer has proved most satisfactory for titration of the microbiological assays. The titrations are done directly in the culture tubes. The contents are stirred by a small stream of air.

### Conclusion

In conclusion, the method that is outlined above has been used in this laboratory, and the Cannon Automatic Dispenser and Titrator has proven very satisfactory. The results that have been obtained for the methionine content of various proteins have compared favorably with those quoted in literature.

# A PRACTICAL THEORY OF COPOLYMERIZATION

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*University of North Dakota*

The process of polymerization is of great importance in the present day chemical industry. This is the name given to the process by which our synthetic rubbers and synthetic plastics are formed from simpler substances. These simpler substances are of low molecular weight, and the polymerization process involves the joining together of these simple compounds called monomers into substances of very high molecular weight. These latter compounds often consist of several hundred or thousand monomer units. If the plastic or rubber polymer is composed of only one kind of monomer, such as styrene or butadiene, then the material is called a simple polymer. If the high molecular weight compound is formed from mixed monomers, it is called a copolymer. Most copolymers are formed from only two principal monomers, and this is the type of copolymerization process with which we are dealing in the present discussion.

A number of theoretical studies have been published by Wall, Lewis, Mayo, Alfrey, and others on the kinetics of copolymerization. Such studies led to the determination of relative reaction rates of various monomers used in copolymerization.

Our present theory is based on the results of actual laboratory experiments on copolymerization. These studies were the outcome of an attempt to learn the reason that a certain copolymer system had led to conflicting and inconsistent results in many studies which had been made on this system. The fundamental relationship on which this empirical theory is based may be stated as follows: The composition of a copolymer forming at any instant is a linear function of the composition of the monomer substance. Stated differently, this means that the fraction of monomer A in the copolymer forming at any instant is proportional to the fraction of A in the monomers.

In the experimental study mentioned, several series of monomer mixtures were prepared. Their compositions varied step-wise from mixtures containing 5% A and 95% B to those containing 95% A and 5% of B. Many samples of each composition were polymerized in otherwise identical recipes and samples were obtained at various stages of conversion, especially low conversions. After chemical analysis to determine the percent of monomer A in each sample copolymer, a graph was constructed showing the percent of A in the copolymer plotted against the percent conversion. Extrapolation of the curve for each original monomer composition to zero percent conversion gave the value of the percent of A in the first copolymer formed.

Next the percentage of A determined as above was plotted against the various original compositions of monomers. A very good straight line resulted. This led to the statement of the relationship given

earlier: The weight fraction of a monomer in the polymer forming at any instant is proportional to the weight fraction of that monomer in the monomer mixture.

### Mathematical Analysis

Let  $a$  = weight of monomer A in the monomers at any time.

$a_0$  = weight of monomer A in the starting mixture.

Similarly:

Let  $b$ ,  $b_0$  represent the same terms for monomer B.

Also for simplicity, assume  $a_0 + b_0 = 1$

Then the weight fractions of A and B in the monomers at any time are, respectively:

$$\frac{a}{a + b} \quad \text{and} \quad \frac{b}{a + b}$$

Now, representing the quantities of monomers A and B undergoing polymerization at the instant in question by  $da$  and  $db$ , the weight-fraction of monomer A in the polymer being formed is

$$\frac{da}{da + db}$$

Our fundamental relationship stated earlier then becomes

$$\frac{da}{da + db} = (\text{constant}) \left( \frac{a}{a + b} \right)$$

where the constant equals the slope of the graph of monomer-polymer compositions.

Rearranging and integrating

$$\ln a = (\text{constant}) \ln (a + b) + \text{constant}$$

Evaluating the integration constant and changing to common logarithms:

$$\log \frac{a}{a_0} = (\text{constant}) \log (a + b)$$

This expression and the differential equation from which it was derived serve for several calculations which together give us a very complete picture of the copolymerization process involving two monomers, insofar as compositions are concerned.

These expressions can easily be used to make three types of calculations, as follows:

1. To find the average composition of the copolymer formed at any degree of conversion.

2. To find the composition of the copolymer formed at any degree of conversion.
3. To find the degree of conversion at which the faster-polymerizing monomer is used up.

A practical basis for study of many copolymer systems has been described. Considerable experimental work with several copolymer systems has given substantial agreement with this analysis. Furthermore, this theory gives a reasonable interpretation to the observed variability of properties of copolymer systems with degree of conversion.

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## WATER AS AN INTRICATE COMPONENT OF PAINTS

*Wouter Bosch, R. W. Jepson, and L. K. Bjelland*

*North Dakota Agricultural College*

Much work has been done on the utilization of water in the preparation of emulsion paints and various brands of these paints are on the market. These paints, however, are of the oil in water emulsion type which means that they can be thinned with water to give them brushing or spraying consistency. Contrary to this, little attention has been given in this country to WO emulsion paints in which the paint vehicle is the continuous phase and water is present in the dispersed form.

Particularly in Germany prior and during the second world war, considerable research was done on this subject with the intention to make substantial savings on valuable oils and other vehicle raw materials. Hans Wagner and his collaborators have published a rather continuous series of articles on WO emulsion paints in the *Farben Zeitung* from 1936 - 1942 and one of us has prepared some interesting paints of this type in Holland during the war. Wagner showed the important difference between the following 3 types of WO emulsions:

1. Zero emulsions, in which no emulsifying agent is used.
2. Stable emulsions, that contain rather large amounts of emulsifiers.
3. Borderline emulsions, in which homeopathic quantities of emulsifying agents are employed.

The last type is of essential interest as it appears that a portion of the water is chemically bound in the paint or varnish and does not evaporate upon drying or even on baking at 120° C. for one hour. Many properties, such as drying time, gloss, hardness, flow, brushability, weather resistancy, of paints made from WO emulsion vehicles show improvements over those of paints made from the same vehicles without the use of water.



## BRUCELLOSIS IN NORTH DAKOTA SWINE

*Alice I. Goldsby**Department of Veterinary Science**North Dakota Agricultural Experiment Station*

Brucellosis in swine was first described by Traum.<sup>1</sup> Since the original description of the disease there have been numerous reports of the prevalence of swine brucellosis and at the present time there are numerous investigations in progress concerning the economic and public health aspects of swine brucellosis.

It has been quite generally accepted that the swine organism *Brucella suis* is quite pathogenic to man. Evidence also indicates that *Brucella suis* may be pathogenic to cattle.

During the past year or so the livestock producers, public health authorities and veterinarians have been summarizing and reevaluating the existing information on the general problem of brucellosis. The ultimate aim is the complete eradication of this disease.

The diagnosis of brucellosis is based on isolation of the organism from blood or tissues of suspected cases or by the agglutination method. Crawford and Manthei<sup>2</sup> regard an agglutination titer of 1:100 as indicative of brucella infection in swine. It has, however, been shown in numerous investigations that titers of 1:50 or 1:25 may be found in swine with active brucellosis.

In a previous study it was found that North Dakota white tailed deer appear to be very resistant to brucellosis since only 0.22% showed a significant agglutination titer.<sup>3</sup>

The North Dakota Livestock Sanitary Board recently passed a regulation requiring breeding swine to pass a negative brucellosis test before entrance into the state.

It seemed that a survey of the incidence of brucellosis in North Dakota and western Minnesota swine would furnish information of value in the preliminary phase of a program directed against swine brucellosis.

Blood samples of swine were collected on the killing floor of the Armour and Company packing plant at West Fargo. One day's kill was selected as a more or less representative sample of the swine marketed. The serum agglutination test was conducted using U.S.D.A. Bureau of Animal Industry antigen.

Five-hundred-forty-three samples of blood were collected and the serum tested at a 1:25 dilution. One-hundred-twenty-one or 22.3% were either positive or suspicious for the presence of brucella agglutinins. One-hundred-six of these positive sera were retested at

<sup>1</sup> Traum, Jacob, Report of the Chief of the Bureau of Animal Industry, U.S. Department of Agriculture, page 30 (1914).

<sup>2</sup> Crawford, A. B. and Manthei, C. A., Brucellosis of Swine, U.S.D.A. Circular 781 (1948).

<sup>3</sup> Bolin, F. M., Goldsby, Alice I., Rheault, Pat and Eveleth, D. F., Brucellosis in North Dakota Deer, North Dakota Agricultural Experiment Station Bimonthly Bulletin, Volume XI, No. 3, pages 102-103 (1949).

various dilutions. The results are as follows: Of the 106 samples 90 were at least suspicious in the 1:50 dilution and 39 were positive or suspicious in the 1:100 dilution and 10 were positive or suspicious in the 1:200 dilution. At the 1:100 dilution 7.4% of the samples showed complete or partial agglutination.

These data collected on only a small number of swine indicate that 7.4% of the swine killed in a single day at the West Fargo packing plant may be suspected as being infected with brucella organisms.

The incidence of swine brucellosis found in this study is higher than that reported for cattle in the state.<sup>4 5 6</sup>

The findings in this investigation indicate that a vigorous program for the eradication of swine brucellosis is needed.

### Summary

Swine killed at the West Fargo packing plant of Armour and Company showed agglutination titers of 1:100 or higher in 7.4% of the animals.

The author is indebted to the officials of Armour and Company and to Dr. J. L. Meyers for assistance in making this study. To Mrs. Pat Rheault and Mrs. Muriel Stringer are due thanks for collecting the blood and aid in completing the tests.

## THE TOXICITY OF NITRATES FOR BABY CHICKS

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Poisoning of chickens by potassium nitrate was investigated by Guberlet<sup>1</sup> who found that the toxic dose was about one gram of the salt per pound of body weight. In these studies single large doses of potassium nitrate in water were given. This investigator lists thirst, anorexia, vomition, diarrhea, retarded heart action, subnormal temperature and cyanosis as symptoms.

Biester and Schwarte<sup>2</sup> in summarizing the literature on sodium chloride poisoning in birds list loss of appetite, severe depression and progressive paralysis followed by respiratory failure as the outstanding symptoms.

<sup>4 5 6</sup> Brandenburg, T. O., Report of the North Dakota Livestock Sanitary Board (1946-47-48).

<sup>1</sup> Guberlet, John E., Potassium Nitrate Poisoning in Chickens, with a Note on its Toxicity, Journal of the American Veterinary Medical Association, Vol. 62, pp. 362-365 (1922).

<sup>2</sup> Biester, H. E., and Schwarte, L. H., Diseases of Poultry, Second Edition (1948).

Abbott<sup>3</sup> has reported on the high nitrate content of well water of some North Dakota wells and pointed out the danger of the use of such water in infant feeding.

The formation of methemaglobin by nitrites produced in the gastrointestinal tract from nitrates has been stressed by numerous investigators of nitrate poisoning in mammals. Guberlet does not mention methemaglobin in his report on nitrate poisoning in chickens.

In some flocks of chickens there are yearly losses of young chicks from unknown causes. It appeared worth while to determine if some of these losses could be due to nitrate poisoning.

In the first trial (Table I) day old chicks were given a standard growing ration containing 1% added sodium chloride. The drinking water was distilled with or without added sodium nitrate or sodium chloride. In these trials all of the chicks did well in the ten day trial except those receiving the water containing 1% sodium chloride. Eighteen of the twenty birds used died with a generalized edema.

Since in this trial there was no evidence of toxicity from the nitrates a second trial was made (Trial II, Table I).

In this case the deaths in the two groups receiving 0.073 molal sodium chloride and sodium nitrate were nearly the same and both groups presented similar pathology.

Chicks dying from both sodium chloride and sodium nitrate showed a generalized edema with or without a deposition of urates on the visceral organs. In neither case was there evidence of methemaglobin formation.

Since chicks did not appear to be capable of reducing nitrates to nitrites and since sodium chloride was equally as toxic as sodium nitrate a different explanation of these death losses was needed.

It has been shown by Scrivner<sup>4</sup> and confirmed by us<sup>5</sup> that by decreasing the "salt" intake of chicks and poults that the incidence of generalized edema can be decreased.

There are also certain similarities in the generalized edema from kidney damage in birds to the formation of urinary calculi in mammals which may or may not cause edema. Some animals often show edema but calculi cannot be grossly demonstrated. Beeson, Pence and Holm<sup>6</sup> have shown that in many cases the primary calculi are

<sup>3</sup> Abbott, G. A., High Nitrates in Drinking Waters, and Their Toxicity to Infants, Proceedings of the North Dakota Academy of Science, Vol. 2 (1949).

<sup>4</sup> Scrivner, L. H., Experimental Edema and Ascites in Poults, Journal of the American Veterinary Medical Association, Vol. 108, pp. 27-32 (1946).

<sup>5</sup> Eveleth, D. F., Bolin, F. M. and Goldsby, Alice I., "Mushy" Chick or Poult Disease, N. Dak. Agricultural Experiment Station Bimonthly Bulletin, Vol. 9, No. 6, pp. 159 (1947).

<sup>6</sup> Beeson, W. M., Pence, J. W. and Holm, G. C., Urinary Calculi in Sheep, American Journal of Veterinary Research, Vol. 4, No. 11, pp. 120-126 (1943).

formed in the kidney tubules of lambs. This appears to be identical to the condition in fowls where uric acid deposits form in the tubules and prevent water elimination thus causing edema.

Cases observed in post mortem examination of "visceral gout" present deposits of uric acid on the visceral organs without edema. It is theorized that the deposition of uric acid in tissues is the result of a failure in kidney function, but which in absence of excessive sodium salts does not produce an effusion of fluid into the tissues.

### Summary

Water containing 4000 PPM of nitrate is nontoxic to day old chicks. Water containing 0.073 molal concentrations of either sodium chloride or sodium nitrate has a tendency to produce kidney damage and generalized edema in chicks.

**TABLE I.**

**Toxicity Trials with Sodium Nitrate and Sodium Chloride**

<b>Trial I.</b>				
	<b>Initial No.</b>	<b>Final No.</b>	<b>Initial Weight</b>	<b>Final Weight</b>
NaCl 1% .....	20	2	41.5	Edematous
Distilled water	20	20	41.4	94.1
NO <sub>3</sub> —				
100 PPM .....	20	20	42.3	90.5
250 PPM .....	20	18	41.2	83.2
500 PPM .....	20	18	40.1	86.2
1000 PPM .....	20	20	41.0	84.7

### Trial II.

**Comparison of Toxicity of Sodium Nitrate and Sodium Chloride**

	<b>Initial No.</b>	<b>Final No.</b>	<b>Initial Weight</b>	<b>Final Weight</b>
Distilled water	10	10	36.4	98.2
*NaCl .43%	10	7	34.4	102.2
NO <sub>3</sub> —				
1:2000 PPM .....	15	14	34.3	89.3
*1:4000 PPM .....	15	12	36.2	93.5

\* 0.073 molal solutions.

## POST MORTEM EXAMINATION OF NORTH DAKOTA DEER

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In an earlier report we summarized the results of agglutination tests on deer blood for the detection of brucellosis.<sup>1</sup> These tests showed that only 0.22% of the deer tested were possibly infected with brucellosis.

This report summarizes the results of post mortem examinations of deer carcasses and viscera made during 1947 and 1948. During the hunting season of 1947 deer killed in the Lower Souris Game Refuge were examined at the checking station. No actual count was made of the carcasses examined but a conservative estimate would be 300. In no case was evidence of disease observed. The deer were fat and free of external parasites. The viscera of 86 of these deer were later examined at the laboratory.

During the late winter of 1947-48 an additional number of deer carcasses were examined. As reported by McKean<sup>2</sup> most of these died as a result of malnutrition and predatory animal attacks. In the carcasses examined there was no evidence of an infectious disease contributing to the death of the deer.

During the 1948 hunting season a more complete study of the visceral organs and in some cases the carcasses was made. Most of the carcass examinations were made at the Dawson Game Refuge. Here approximately 50 carcasses were examined. No gross evidence of any infectious disease was found. One rather emaciated carcass was observed, but in this case the animal was an old buck that had lost his teeth.

All or portions of the viscera from 425 deer have been examined in the laboratory phase of this study. These viscera were identified in some cases, as to sex and area where killed or had died. The organs present were usually the heart, lungs, liver, spleen, pancreas, lymph nodes and all or parts of the urinary and reproductive systems. In Table I a minimum number of the various organs examined is given. This number is a minimum because many carcasses in which the organs of the thoracic cavity were present were examined but not tabulated.

The information obtained shows that such chronic diseases as tuberculosis, caseous lymphadenitis, chronic pasteurellosis nor any of the acute septicemic diseases are common in North Dakota deer.

<sup>1</sup> F. M. Bolin, Alice I. Goldsby, Pat Rheault and D. F. Eveleth—Brucellosis in North Dakota Deer, Bimonthly Bulletin, Vol. XI, No. 3, (1949).

<sup>2</sup> McKean, W. T.—Personal Communication.

The importance of these findings may be stated in two ways. First, the deer in North Dakota are very healthy. This should insure relatively low losses from disease and also insure a larger yearly hunting kill of normal animals. Secondly, the absence of any degree of infections by the serious diseases in the deer insures little or no possibility of the deer transmitting diseases to domestic livestock or man.

In two cases the kidneys of the deer were studded with small necrotic foci. From both specimens similar organisms probably *Corynebacteria* sp. were isolated.

The general health of the deer examined is equal to or better than the average fat animal killed in our inspected abattoirs.

From the data obtained it would appear that there is a much greater danger of transfer of disease from domestic livestock to deer than there is from deer to domestic livestock.

### Summary

There is no evidence that the white tailed deer of North Dakota act as a reservoir of infection of those chronic or acute infectious diseases transmittable to domestic livestock.

The authors are indebted to the hunters, wild life clubs and members of the Federal Aid Division of the North Dakota Game and Fish Department for aid in the collection of data in this study.

The author also wishes to express his appreciation to the other members of the staff of the Department of Veterinary Science of the North Dakota Agricultural College for assistance in examining the carcasses and visceral organs discussed in this report.

**TABLE I.**  
**Number of Various Organs Examined**

	Hearts	Lungs	Livers	Kidneys	Spleen	Testicles	Ovaries	Rumens	4th Stomach	Small Intestines	Large Intestines	Total No. Deer Observed	Females	Males	Unknown
Hunting Season Deer, 1947	7	15	4	25				79	81	80	81	86	14	7	65
Twelve Starved Deer from Arrowwood, 1947	12	12	12	12	12			12	12	12	12	12			12
Hunting Season Deer, 1948	78	148	146	231	213	7	41	314	301	313	306	326	161	145	20
<b>TOTAL</b>	<b>97</b>	<b>175</b>	<b>162</b>	<b>268</b>	<b>225</b>	<b>7</b>	<b>41</b>	<b>405</b>	<b>394</b>	<b>405</b>	<b>399</b>	<b>424</b>	<b>175</b>	<b>152</b>	<b>97</b>

## DEGREE-DAYS FOR HEATING AND COOLING IN NORTH DAKOTA

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*North Dakota Research Foundation*

The North Dakota Research Foundation is preparing a new bulletin entitled, "Industrial Development in North Dakota—Resources and Opportunities." In this bulletin is being included a section containing basic meteorological data necessary in considering North Dakota as a possible location for industry. One such item of information being included is a tabulation of the degree-days of heating and of cooling for the following eight points in North Dakota: Bismarck, Devils Lake, Dickinson, Fargo, Grand Forks, Jamestown, Minot, and Williston.

A degree-day of heating may be defined "as one on which the mean temperature is 1° below 65°." In a similar way, a degree-day of cooling may be defined as one on which the mean temperature is 1° above 75°. It has been established that the total heating load or cooling load on a given structure in a given location is quite closely proportional to the degree-days of heating or of cooling, as the case may be. Therefore, a knowledge of the degree-days in any location is of prime importance to the architect in designing a building and in determining the amount of insulation and type of construction. It is also just as important to the heating and ventilating engineer in designing the heating system and in estimating its cost of operation. With increased interest in air conditioning it is also desirable to use the concept of degree-days of cooling as a measure of the work to be done in maintaining reasonable temperature during the warmer months of the year. In view of these uses, it is desirable that precise data, fairly representing average conditions, be available.

In an effort to provide sound data for selected points distributed throughout the state, the North Dakota Research Foundation has computed the degree-days of heating and cooling for Bismarck, Devils Lake, Dickinson, Fargo, Grand Forks, Jamestown, Minot, and Williston. The following table presents a summary of the data so calculated.

This table is based upon the data for thirteen years, 1935-47, inclusive, which includes the hottest and coldest years on record. In computing degree-days the daily deviation from 65° for heating and 75° for cooling was summed to give the degree-days for that particular month. The sum of the monthly figures gave the total for the year. For the 13-year period the averaging was done by months. The detailed data is available in the files of the Research Foundation.

<sup>1</sup> GAS ENGINEERS' HANDBOOK, First Edition. McGraw-Hill Book Company, p. 905 (1934).

**DEGREE-DAYS — HEATING AND COOLING**  
1935 - 47 Average

**HEATING**

City	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Total
Bismarck	1700	1520	1150	652	329	121	12	33	229	556	1130	1460	8,890
Devils Lake	1870	1650	1290	751	406	156	23	58	294	624	1240	1620	9,980
Dickinson	1690	1520	1210	723	413	185	30	65	291	624	1170	1440	9,360
Fargo	1790	1580	1200	694	339	104	13	36	241	567	1140	1560	9,260
Grand Forks	1850	1640	1270	727	381	121	16	47	288	613	1210	1630	9,790
Jamestown	1770	1570	1190	706	354	121	15	65	257	577	1150	1520	9,300
Minot	1760	1570	1230	723	375	161	21	57	268	580	1200	1520	9,460
Williston	1670	1520	1180	663	340	143	11	53	250	565	1150	1440	8,980

**COOLING**

City	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Total
Bismarck	0	0	0	0	0	8	58	36	6	0	0	0	108
Devils Lake	0	0	0	0	1	3	33	16	1	0	0	0	54
Dickinson	0	0	0	0	0	5	30	17	1	0	0	0	53
Fargo	0	0	0	0	0	5	45	30	5	0	0	0	85
Grand Forks	0	0	0	0	2	4	35	20	2	0	0	0	63
Jamestown	0	0	0	0	1	6	66	29	4	0	0	0	106
Minot	0	0	0	0	1	6	31	18	3	0	0	0	59
Williston	0	0	0	0	1	8	44	21	3	0	0	0	77



An analysis of the data given shows that, in general, the northern and eastern cities have a greater need for heating in the winter than do the southern and western. It is also evident that the range of degree-days of heating is less in the western cities than in the eastern. This indicates that the temperatures in the western part of the state are not only milder than in the eastern part of the state, but more uniform.

The south-central cities of Jamestown and Bismarck have the greatest need for cooling in the summer months, such a need decreasing as you pass either to the east or west or north. These same cities have a greater range of degree-days of cooling, therefore possessing, not only higher mean temperatures, but a greater variation.

It is well worthy of note that every month in the year possesses some degree-days of heating but that only five months of the year possess any degree-days of cooling. This is understandable when it is remembered that North Dakota has the lowest mean annual temperature, approximately 41° F., of any state in the Union. It is surprising to note the low total degree-days of cooling, especially as one remembers the days of July and August. This may be somewhat misleading until one remembers that the degree-days are based upon the daily average temperature; and even though summer days may be hot in North Dakota, the nights are invariably cool.

There are now available precise tabulations of degree-days of heating and cooling for eight cities distributed throughout the state and based upon average conditions, including coldest and hottest years, for thirteen years.

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## SIDELIGHTS ON THE WHEAT STEM SAWFLY, CEPHUS CINCTUS NORTON

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One of the most destructive insect pests of wheat in North Dakota is the wheat stem sawfly. It was first recorded from native grasses in 1890 and was named the grass stem sawfly. At that time it was not known to attack small grain. However, the following prophetic statement was then made: "The economic importance of this species arises from the fact that it may be expected at any time to abandon its natural food-plant in favor of small grains on which it can doubtless successfully develop."

In 1895, only five years later, James Fletcher, then entomologist for the Dominion of Canada, first announced its destructive attack on wheat in Manitoba. Since that time, as the acreage of native grasses has been decreased by bringing wild land under cultivation the sawfly has been shifting its destructive feeding more and more to wheat and to some extent other small grains. The

area of sawfly damage has become increasingly extensive. It occurs widely in North Dakota, Montana and the adjoining Prairie Provinces of Canada. Heaviest damage in North Dakota occurs in the northwestern third of the state where sawfly has caused an approximate loss of from 2½ million to 5 million bushels of wheat annually for the past eight years. In Montana the loss has been approximately the same. In the prairie section of Canada where the sawfly's changing food habit, from grass to wheat, was first recorded it is responsible for the loss of millions of bushels of wheat annually.

Part of this loss is due to the feeding of the sawfly larvae in the developing stems. Such activity causes shrinkage of the kernels and lowered productivity of the heads. Investigations conducted by the North Dakota Agricultural College Experiment Station have revealed that the sawfly shows a decided preference for the more vigorous, high-yielding stems and reduces their yield nearly 10 percent. The magnitude of this type of loss had not been previously realized.

An additional type of loss which has long been known is due to the breaking over of stems that have been mined and weakened by the larvae. As the grain is nearing the ripening stage, the larva which has bored downward within the stem, reaches the base where it cuts a V-shaped groove completely around the inside of the stem at the ground. The larva then retreats to the base and plugs the stem with its chewings directly beneath this groove. It is thus well protected against the weather and most other hazards. The stem breaks over at the groove to provide an escape for the insect after it transforms to the adult, winged stage the following spring. Burning of dry stubble fields is of no avail in control after the larva has retreated to its hibernating place in the base of the stem.

The amount of grain lost in fields by sawfly will vary largely according to the degree of infestation, the delay in harvesting, the amount of breaking over as influenced by rain and high winds prior to harvesting, the density of the stand, and the variety of wheat grown.

The adult sawflies emerge from the stubs of grasses and small grains in June. After mating, the females lay their eggs within the stems of the developing plants. She does this by means of a sawlike apparatus located at the tip of her abdomen which she thrusts into the stem to deposit the egg. A female will normally lay about 50 eggs and usually but one to a stem. Upon hatching, which requires one week, the larvae begins feeding within the stem as it bores its way downward to reach the base before the grain is ripe.

On the basis of recent investigations conducted by the North

Dakota Agricultural Experiment Station it has been found that the application of insecticides as yet developed, are impractical in control of the wheat stem sawfly.

The early application of shallow tillage which exposes the stubble as well as the roots to weathering is highly effective in destroying the sawfly larvae.

Sawflies not only show a decided preference for the more vigorous and high yielding stems but also reduce their yield nearly ten per cent due to shrinkage of the developing kernels. This shrinkage is a type of loss which is usually disregarded because it is not as spectacular as the commonly recognized loss resulting from the breaking over of the infested stems.

Wheat may be protected against sawfly by delayed seeding but to what extent this "advantage" might be offset by the decreased yield from late seeding should be determined.

The parasite, *Microbracon cephi* may be largely responsible for the shifts of intensity and changes in sawfly populations.

Early harvesting, before storms cause the infested stems to break over, is at present the most practical means of avoiding loss from fallen heads.

Rescue is the only commercial hard spring wheat with much resistance to sawfly. Some of the durums now being generally grown are also very resistant.

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## VOLATILE PRODUCTS FROM THE PREHEATING OF PHENOL-FORMALDEHYDE RESINS

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Recent studies by Eells and Dunbar<sup>1</sup> have emphasized the significance of moisture in preheating phenolic molding powders. They have shown that steam preheating produces molded articles of as good general quality as dry preheating, but greatly reduces the time required for molding operations. Any attempt to explain this behavior would of necessity involve a qualitative and quantitative investigation of the volatile products evolved during preheating. At the present time, little mention is found in the literature concerning the volatiles that are liberated during the preheating and molding of phenolic resins. Redman and others<sup>2</sup> made rather detailed studies of the initial condensation between phenol and hexamethylenetetramine in the dry state by means of the volatiles evolved. Williamson<sup>3</sup>

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<sup>1</sup> D. E. Eells and R. E. Dunbar, Proceedings of the North Dakota Academy of Science, 1, 37, (1948).

<sup>2</sup> L. V. Redman et al., Ind. Eng. Chem., 6, 3, (1914).

<sup>3</sup> R. V. Williamson, Modern Plastics, 25, 126, (1948).

attempted quite successfully to correlate the total volatile loss in molding phenolics with preheat time, and molding time and temperature. We have recently undertaken a series of studies involving the volatile products liberated under varied conditions of preheating. Phenol, ammonia and water are the most important products liberated.

The investigation was conducted using standard disc preforms of BM120 with an average mass of 22 g. The initial preform moisture content was determined by the Dean-Stark method,<sup>4</sup> and the preforms were then stored at constant humidity until used. The rates of heating under varied conditions of oven temperatures, preheat temperatures and added moisture were investigated in order to find the relationships involved.

The preheating oven was arranged in such a manner as to simulate as nearly as possible the actual preheating conditions employed in industry at present. Preheat temperatures of 160°, 180° and 210° F., and moisture conditions of 0, 40 and 80 drops per minute for each preheat temperature were selected for consideration. The oven was equipped with a pressure chamber into which the samples were placed for preheating. Moisture could be introduced at the desired rate from a constant head reservoir. The chamber was swept during preheating at the rate of one liter every eight seconds, and the volatiles were collected in 0.2 M sulfuric acid. The air entering the oven was directed first through concentrated sulfuric acid to remove moisture and ammonia, then through an ascarite tube to remove carbon dioxide and acid fumes, and finally through a hot copper tube preheater. The use of the preheater reduced the drop in oven temperature during sweeping.

Samples of about 110 g. were placed in the oven and preheated for the desired time under the conditions selected. The loss in weight was recorded after each run. The residual moisture in the preforms was determined only after the dry preheats. It would be meaningless for our purposes to perform this operation after steam preheating as the preforms absorb moisture from the oven in such cases. Qualitative and quantitative tests were then made on the acid solution used for volatile collection. The phenol was determined gravimetrically as tribromophenol and colorimetrically by Mumford's method.<sup>5</sup> Ammonia was determined by the micro-Kjeldahl method. Tests for the presence of methylamine and formaldehyde were negative. The quantity of ammonia decreased with the addition of water to the oven until a fairly constant value was reached, and then the addition of more water produced little or no change. The quantity of phenol, however, decreased steadily with added moisture. Extended dry preheats indicated that the quantities of both phenol and am-

<sup>4</sup> A. S. T. M. Committee D-1, A. S. T. M. Standards on Paint, Varnish, Lacquer, and related products; American Society for Testing Materials, Philadelphia 3, Pa., p. 559 (Dec. 1947).

<sup>5</sup> E. M. Mumford, Chem. News, 107, 253, (1913) cf. C. A., 7, 2734, (1913).

monia liberated were still increasing after 1½ hours. These extended preheats were accompanied by an appreciable drop in oven temperature.

The absence of formaldehyde from the volatiles collected is significant, indicating that the condensation between the novolac and formaldehyde is extremely rapid. The fact that formaldehyde is actually formed as such might appear questionable. However, we have found that under certain conditions the preforms actually gain weight on a dry basis. That is to say that the measured volatile loss during the preheating exceeds the weight loss. This is most pronounced in the cases in which sweeping is not attempted. This is probably due to the more constant oven temperature. The only explanation that we are prepared to offer at the present time is that oxygen from the water in the preforms is being added to the resin in the form of methylol groups supplied by the formation and subsequent condensation of formaldehyde. This behavior was noted while preheating to 210° F. under dry oven conditions, and indicates the breakdown of the hexamethylenetetramine into ammonia and formaldehyde through interaction with the water originally present in the preforms.

### Conclusions

The results of this study indicate that:

- (1) The curing reaction is initiated in the preheating stage.
- (2) The introduction of moisture into a preheating oven causes a reduction of the volatile products liberated, and hence a reduction in the degree of crosslinking.
- (3) Preform moisture reacts to some extent with the hexamethylenetetramine to produce ammonia and formaldehyde.

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## WATER REQUIREMENTS FOR INDUSTRY

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Water is one of the essential raw materials in industry. It is just as much a raw material as is iron ore, coal, lime, or sulfur; and its availability in sufficient quantity, and of satisfactory quality and temperature is of paramount importance. Water supply is always considered in deciding upon the location of industries. In some industries it may be the determining factor; in all industries it is of importance.

The quality requirements for industrial waters, while well known, are not as important as quantity requirements in locating industries, because the modern water technologist can tailor any given water to any given set of specifications. Therefore, quality requirements are not touched upon. Temperature requirements, particularly of water for process operations and cooling and condensing, are important. A later paper will discuss the temperature of North Dakota waters.

This paper deals only with the quantity requirements. Industries require water for the following major uses:

1. Steam raising.
2. Process operations.
3. Cooling and condensing.
4. Fire protection.
5. Sanitary needs.
6. Waste disposal (dilution).

No effort has been made to distribute the water requirements among the various uses. The figures reported are gross figures for the first five uses. No water is included for waste disposal.

**TABLE I.**  
**Water Requirements for Selected Industries**

Industry	Water Requirement gal. / ton
1. Ammonia .....	31,000
2. Ammonium suphate	200,000
3. Beet sugar	2,600- 3,200 beets 20,000-25,000 sugar
4. Blast furnace	4,000
5. Carbon dioxide .....	23,000
6. Caustic soda .....	21,000
7. Cellulose nitrate	10,000
8. Coal carbonizing .....	3,500
9. Coal washing .....	125
10. Corn refining .....	333
11. Electric plant	120,000
12. Glycerine .....	1,100
13. Gun Powder	200,000
14. Hydrogen .....	660,000
15. Industrial alcohol .....	20,000
16. Lactose .....	220,000
17. Magnesium carbonate	39,000
18. Paper	
a. De-inking paper	38,000
b. Paper board	14,000
c. Strawboard .....	26,000
19. Phosphoric acid	7,500
20. Rock wool .....	5,000
21. Steel plant .....	20,000-35,000
22. Soda ash .....	18,000
23. Sulfur mining	3,000
24. Tanning	16,000

**TABLE II.**  
**Water Requirements for Selected Industries**

<b>Industry</b>	<b>Water Requirement gal. / 100 lbs.</b>
1. Cotton	
a. Processing	3,800 goods
b. Dyeing .....	1,000-2,000 goods
2. Linen .....	10,000 thread
3. Milk and milk products	
a. Butter	250
b. Cheese	220
4. Rayon	
a. Dissolving pulp .....	9,500 pulp
b. Cupra-ammonium yarn .....	8,000 yarn
c. Viscose yarn	10,000 yarn
5. Vegetable dehydration	
a. Beets .....	1,870
b. Cabbage	770
c. Carrots .....	1,580
d. Potatoes	560-1,250
e. Rutabaga .....	1,520
f. Sweet potato .....	900
6. Wool	
a. Scouring	2,000-15,000 raw wool

These figures are not mathematical averages, nor are they necessarily typical. They do, however, represent the quantity requirements of operating plants (except for waste disposal). Requirements for a given plant, new or old, will depend upon design and water economy, and may vary greatly from the figures given. These data should be used with caution, and their main value lies in emphasizing the quantity requirements of industry.

Industries require water in varying quantities, depending upon the process, the plant design, and the economy of water utilization. The quantities required are frequently enormous in comparison with the plant output. The figures cited in Tables I, II, and III have been obtained from a variety of sources; some from incidental references in the literature, some from correspondence, and some from plant inspections. In every case the experience of at least three plants is involved in arriving at a cited value.

From the data presented, it is apparent that water is one of the essential raw materials of industry, even on a tonnage basis. The availability of water in sufficient quantity is one of the major factors in plant location; it may determine whether or not a certain industry is located in a certain community, or whether, if so located, it succeeds. This has a direct bearing on the location of industries in North Dakota. Some communities, amply supplied with other raw materials, lack supplies of industrial water. Past experience as well as reflection would show the folly of locating an industry without regard to the water supply. On the other hand, there are places in the state with ample water supply, and the development of the Missouri River Basin projects will add to the number of these sites.

TABLE III.

## Water Requirements for Selected Industries

Industry	Water Requirement
1. Air Conditioning	6,000-15,000 gal. / person / season
2. Canning	
a. Asparagus	7,000 gal. / 100 cases No. 2 cans
b. Beans	
(1) Green	3,500 gal. / 100 cases No. 2 cans
(2) Lima .....	25,000 gal. / 100 cases No. 2 cans
(3) Pork and .....	3,500 gal. / 100 cases No. 2 cans
c. Corn	
Cream or whole .....	4,000 gal. / 100 cases No. 2 cans
d. Peas .....	3,000 gal. / 100 cases No. 2 cans
e. Sauerkraut .....	300 gal. / 100 cases No. 2 cans
f. Spinach	16,000 gal. / 100 cases No. 2 cans
g. Tomatoes	
(1) Products	7,000 gal. / 100 cases No. 2 cans
(2) Whole .....	750 gal. / 100 cases No. 2 cans
3. Meat Packing	
a. Slaughter house .....	16,000 gal. / 100 hog unit
b. Packing house .....	55,000 gal. / 100 hog unit
c. Poultry	220 gal. / 100 lb. live weight
4. Milk and milk products	
Receiving and bottling ...	450 gal. / 100 gal. milk
5. Oil fields	18,000 gal. / 100 bbl. crude oil
6. Oil refinery	77,000 gal. / 100 bbl. crude oil



## SUGGESTIONS TO AUTHORS

**General.** Authors should submit to the secretary of the Academy at the time of the annual meeting following presentation of the paper two copies of the manuscript typed double space with wide margins on 8½" by 11" white paper.

At present it is impossible to include colored drawings, photographs, or elaborate figures.

**Titles and Footnotes.** Titles should be specific but should be kept short by deleting unnecessary words. The title should also show the name and address of the author's institution. Additional footnotes showing other pertinent information are desirable although not obligatory.

**Abstract.** A concise abstract of about 200 words must be supplied to the secretary of the Academy before the annual program is arranged. In case the complete manuscript is not published in the Proceedings the abstract as submitted will be used. The abstract should state the principal result and conclusions and should contain, largely by inference, adequate information on the scope and design of the investigation. The abstract in general should conform to the suggestions outlined for complete manuscripts.

**Literature.** In general only recent papers need be listed and these can often be cited more advantageously throughout the text than in the introduction. Long introductory reviews should be avoided especially when a recent review in another paper or in a monograph can be cited instead. References are arranged and numbered in alphabetical order of author's names and show author, title, journal, volume, first and last pages, and year. The list is given at the end of the paper. Reference numbers must invariably be cited in the text, but author's names and year may be cited also. Abbreviations for names of journals follow the list given in *Chemical Abstracts* 40, Pt. 4: I-CCIX. 1946.

**Organization.** The standard organization involves main sections for introduction, materials, methods, results, discussion, summary, and acknowledgements and literature cited. Alternately a group of related studies each made with different materials or methods may require a separate section for each study with sub-sections for materials and methods and for results under each section. Center headings are used for main sections and italicized, run-in headings for sub-sections. Headings should be restricted to these two types only.

**Tables.** Data should be arranged to facilitate the comparisons readers must make. Tables should be kept small by breaking up large ones if this is feasible. Only about eight columns of tabular matter can be printed across the page. Authors should omit all unessential data such as laboratory numbers, columns of data that show no significant variation, and any data not discussed in the text. A text reference can frequently be substituted for columns containing only a few data. The number of significant figures should be minimized. Box and side headings should be kept short by abbreviating freely; unorthodox abbreviations may be explained in footnotes, but unnecessary footnotes should be avoided. Leader tables without a number, main heading, or ruled lines are often useful for small groups of data.

Tables should be typed on separate pages at the end of the manuscript, and their position should be indicated to the printer by typing "(TABLE I)" in the appropriate place between lines of the text.

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