reversed by boiling them for two minutes after making 3 *N* with hydrochloric acid.

OBERLIN, OHIO

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[CONTRIBUTION FROM THE BAKER LABORATORY OF CHEMISTRY AT CORNELL UNIVERSITY]

# HYDRONITRIC ACID AS PRODUCT OF DESOLVATION OF AMMONONITRIC, HYDRAZONITROUS AND AMMONOHYDRAZONITROUS ACIDS. EXPERIMENTAL FORMATION OF SODIUM TRINITRIDE BY AMMONOLYSIS OF SODIUM NITRATE WITH SODIUM AMIDE[1]

BY A. W. BROWNE AND FRANK WILCOXON

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It was first suggested by Turrentine[2] that hydronitric acid, an "ammonated nitride of nitrogen," may be regarded as the analog in the well-known ammonia system of compounds,[3] of nitric acid, a hydrated oxide of nitrogen, in the water system. This analogy has been recognized by Franklin, who uses the name ammononitric acid for hydronitric acid and who has submitted various lines of experimental evidence[4] in support of the idea. From a slightly different viewpoint, workers in this Laboratory[5] have long regarded the trinitrides, or pernitrides, as nitridizing agents analogous to the peroxides, and have considered nitric and nitrous acids, with their chemical aggregates of oxygen and nitrogen atoms, as intermediate in composition and character between hydrogen pernitride and hydrogen peroxide.

Certain experimental evidence seems to indicate that hydronitric acid may be more closely akin to nitrous than to nitric acid. The authors have found, for example, that iodine is not liberated in appreciable amount on addition of 1% solutions of nitric acid to hydriodic acid, although readily liberated by 1% solutions of either nitrous acid or hydronitric acid. Since the latter substance is not theoretically derivable, however, from normal ammononitrous acid, $N(NH_2)_3$, by removal of ammonia, and has never

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[1] This article is respectfully dedicated by the authors to Professor L. M. Dennis, and will be reprinted as Article No. 1 in The Louis Munroe Dennis Quarter Century Volume to be published in 1928 in commemoration of the completion by Professor Dennis of twenty-five years of service as Head of the Department of Chemistry at Cornell University. The current article is based upon the thesis presented to the Faculty of the Graduate School of Cornell University by Frank Wilcoxon in partial fulfilment of the requirements for the degree of Doctor of Philosophy.

[2] Turrentine, THIS JOURNAL, **34,** 385 (1912).

[3] Franklin, (a) *ibid.*, **27,** 820 (1905); (b) *Am. Chem. J.*, **47,** 285 (1912); (c) *Proc. Eighth Int. Cong. Appl. Chem.*, **6,** 119 (1912); (d) THIS JOURNAL, **46,** 2137 (1924).

[4] Franklin, (a) *Science*, **56,** 28 (1922); (b) Ref. 3 d, p. 2142.

[5] Browne and Welsh, THIS JOURNAL, **33,** 1728 (1911). Goldberg, *ibid.*, **34,** 886 (1912). Browne and Hoel, *ibid.*, **44,** 2116 (1922).

yet been obtained experimentally from nitrous acid by the action of ammonia, although readily derivable by deammonation of normal ammononitric acid,[6] $N(NH_2)_5$, it is clearly not to be regarded as an ammononitrous acid, the nitrous acid of the ammonia system.

That hydronitric acid must be in some way related structurally to nitrous acid seems inevitable, however, from the multiplicity of methods for obtaining it from nitrous acid or its derivatives as described by various investigators. These methods include that employed by Curtius[7] in the classical researches that resulted in the discovery of hydronitric acid, that of Thiele[8] and of Stollé,[9] upon which the commercial manufacture of sodium trinitride has been based, and others.[10] All of these methods involve the use of some compound or derivative of hydrazine, a substance shown in this Laboratory[11] to be a dissociating solvent comparable in scope and character with water and ammonia, and like these substances constituting the basis of a system of acids, bases and salts.

Hydronitric acid may therefore be looked upon as *hydrazonitrous acid*, a nitrous acid of the hydrazine system, experimentally obtainable by *hydrazinolysis* of nitrous acid,[12] either in aqueous or in nonaqueous solution, and derivable by *dehydrazination* of normal hydrazonitrous acid,[13] $N(N_2H_3)_3$, as follows.

$$N(N_2H_3)_3 \longrightarrow HN_3\ (+\ 2N_2H_4) \qquad (1)$$

The preparation of sodium trinitride as a product of the ammonolysis of nitrous oxide by sodium amide, either at high temperatures[14] or at low temperatures[15] in liquid ammonia, might be considered to involve initial

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[6] Mendeléeff [*Ber.*, **23**, 3469 (1890)] regarded hydronitric acid as the nitrile of secondary ammonium orthonitrate, $(NH_4)_2HNO_4$. It is now equally obvious that it may be looked upon as the nitrile of primary ammonium hyponitrite, $NH_4ON{:}NOH$.

[7] Curtius, *Ber.*, **23**, 3023 (1890).

[8] Thiele, *Ber.*, **41**, 2681 (1908).

[9] Stollé, *Ber.*, **41**, 2811 (1908).

[10] See for example, Curtius, *Ber.*, **26**, 1163 (1893). Angeli, *Atti accad. Lincei*, [5] **2**, I, 569 (1893). Dennstedt and Göhlich, *Chem.-Ztg.*, **21**, 876 (1897).

[11] Welsh, THIS JOURNAL, **37**, 497 (1915). Welsh and Broderson, *ibid.*, **37**, 816, 825 (1915).

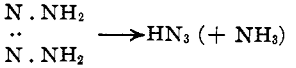
[12] From this viewpoint, reactions involving diazotization of an amine might be regarded as cases of ammonolysis, while those involving diazotization of a hydrazine derivative might be considered instances of hydrazinolysis.

[13] In a preliminary investigation in this Laboratory of the temperature-concentration diagram of the system, $N_2H_4$-$HN_3$, H. E. Riegger obtained evidence, confirmed later by chemical analysis, of the existence of a monohydrazinate of hydrazine trinitride, of the formula, $N_2H_5N_3.N_2H_4$. This white, crystalline compound, the description of which has not yet been published, is at least empirically isomeric with normal hydrazonitrous acid, and may possibly be identical with it.

[14] Wislicenus, *Ber.*, **25**, 2084 (1892). Dennis and Browne, THIS JOURNAL, **26**, 577 (1904).

[15] Joannis, *Compt. rend.*, **118**, 714 (1894). This method of forming trinitrides seems to have escaped the attention of later investigators.

formation of normal *ammonohyponitrous acid*, $N_2(NH_2)_2$, and subsequent deammonation yielding hydronitric acid, as follows.



$$\begin{matrix}\mathrm{N\,.\,NH_2}\\ \cdot\cdot\\ \mathrm{N\,.\,NH_2}\end{matrix} \longrightarrow \mathrm{HN_3}\ (+\ \mathrm{NH_3}) \qquad (2)$$

Numerous organic derivatives of normal ammonohyponitrous acid, known first as the tetrazones,[16] and later as the tetrazenes,[17] have been prepared. Since the decomposition of these tetrazenes has been shown to result in the liberation of nitrogen and the formation of amines,[18] while derivatives of hydronitric acid have not yet been identified among the products of the reaction, it may be more reasonable to consider hydronitric acid from this viewpoint as derivable from isotetrazene, $HN{:}N.NH.NH_2$, which is an empirical isomer of tetrazene, but which cannot like the latter substance be formulated as an ammonohyponitrous acid. Both tetrazene and isotetrazene may, however, be regarded as *mixed ammono-hydrazonitrous acids*.

Derivatives of isotetrazene, variously designated as buzylenes and diazohydrazines, have been prepared.[19] These on decomposition yield derivatives of hydronitric acid and ammonia.

From entirely independent considerations, the formation of hydronitric acid and ammonia by the action upon hydrazine of oxidizing agents that contain no nitrogen, such as hydrogen peroxide, potassium chlorate and potassium persulfate, has been attributed[20] to the intermediate appearance of isotetrazene. On the other hand, tetrazene has been regarded as the probable intermediate product in the case of oxidation by means of potassium permanganate, manganese dioxide, ferric oxide or other oxidizing agents that yield ammonia and nitrogen with little or no hydronitric acid. In terms of the newer nomenclature, therefore, the oxidation of hydrazine may be considered to yield as an intermediate product, under appropriate conditions, either one of the only two theoretically possible ammono-hydrazonitrous acids of the empirical formula $N_4H_4$: namely, tetrazene and isotetrazene.[21]

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[16] (a) Fischer, *Ann.*, **190,** 67 (1878); (b) **199,** 281 (1879). (c) Renouf, *Ber.*, **13,** 2169 (1880). (d) Michaelis, *Ann.*, **252,** 266 (1889). (e) Franchimont and Van Erp, *Rec. trav. chim.*, **14,** 317 (1895). (f) Angeli, *Atti. accad. Lincei*, [5] **9,** II, 180 (1900). (g) Bailey, THIS JOURNAL, **26,** 1006 (1904); (h) **30,** 1412 (1908).

[17] H. Wieland, "Die Hydrazine," Ferdinand Enke, Stuttgart, **1913,** pp. 2, 34, 71, 79.

[18] Ref. 16 b, p. 323. Ref. 16 c, p. 2174.

[19] Curtius, *Ber.*, **26,** 1263 (1893); **29,** 759 (1896). Wohl, *Ber.*, **26,** 1587 (1893). Wohl and Schiff, *Ber.*, **33,** 2741 (1900). Stollé, *J. prakt. Chem.*, [2] **66,** 332 (1902).

[20] (a) Browne, THIS JOURNAL, **27,** 551 (1905). (b) Browne and Shetterly, *ibid.*, **31,** 783 (1909). (c) Browne and Overman, *ibid.*, **38,** 285 (1916).

[21] These are theoretically derivable from the two possible *normal* ammono-hydrazonitrous acids by respective desolvation as follows: $N(N_2H_3)_2NH_2 = N({:}N.NH_2)NH_2 + N_2H_4$, and $N(NH_2)_2N_2H_3 = N({:}\,NH)N_2H_3 + NH_3$.

Further desolvation of the tetrazene may then occur in accordance with the equation $3N\ ({:}N.NH_2)NH_2 = 4NH_3 + 4N_2$.

The iso-tetrazene may undergo similar desolvation as expressed by the equation

From this general viewpoint, all methods that have hitherto been used for the formation or preparation of hydronitric acid may be considered to involve initial solvolysis of an oxyacid of nitrogen or one of its derivatives, with at least potential formation of a normal acid belonging to the ammonia series, to the hydrazine series, or to both, and subsequent desolvation of this acid.

Hydronitric acid is the substance most closely approaching, in composition and character, the *ansolvides* of ammononitric, hydrazonitrous and ammono-hydrazonitrous acids. Complete deammonation of ammononitric acid should yield the anammonide, nitrogen pentanitride, $N^{V}_{3}N^{III}_{5}$, or $N \vdots N(N_3)_2$; while complete dehydrazination of hydrazonitrous acid should result in the formation of the *anhydrazide* $N_3.N_2.N_2.N_3$. The ansolvide of ammono-hydrazonitrous acid would be ordinary molecular nitrogen, if the desolvation involved removal of hydrazine, or the same as that of ammononitric acid if ammonia, only, were removed.

Although these unstable polymeric forms of nitrogen have not as yet been isolated, there is ample experimental proof that both ammonia and hydrazine may be obtained from hydrogen trinitride by direct desolvation with the aid of concd. sulfuric acid.[22] Various investigators in this Laboratory have identified ammonia and hydrazine in the products obtained (1) by passing dry hydrogen trinitride vapor into concd. sulfuric acid,[23] and (2) by bringing together sulfuric acid and ethereal hydrogen trinitride.[24]

## Ammonolysis of Sodium Nitrate

It has been stated by Franklin[4a] that sodium nitrate and sodium amide interact in liquid ammonia when warmed in a sealed tube, yielding sodium trinitride. A further study of this reaction, with especial reference to the behavior of the reagents toward each other at higher temperatures, under ordinary atmospheric pressure, and in the absence of liquid ammonia, has been undertaken by the authors, and constitutes the basis of the present report.[25]

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$N(:NH)N_2H_3 = HN_3 + NH_3$. This would be tantamount to an intramolecular arrangement of the iso-tetrazene to form ammonium trinitride.

[22] Concd. sulfuric acid thus appears to be not merely a powerful dehydrating agent but also in a more general way a strong desolvating agent in the case of solvents of the hydride type. Browne and Overman (Ref. 20 c, p. 293; Ref. 1) have suggested that the formation of ammonia as a product of the Kjeldahl reaction is due to deammonation of the nitrogen compounds by sulfuric acid. The data here cited show the acid to be also capable of effecting dehydrazination.

[23] Helen Isham, Doctor's *Thesis*, "A Contribution to the Chemistry of Hydronitric Acid," Ithaca, N. Y., **1906**.

[24] H. S. Bennett, Doctor's *Thesis*, "The Interaction of Anhydrous Hydronitric Acid and Concentrated Sulfuric Acid," Ithaca, N. Y., **1917**.

[25] Dr. Franklin has very graciously given permission to the authors to investigate this phase of his discovery, and the authors wish to express herewith their deep appreciation of this courtesy and kindly encouragement.

The formation of sodium trinitride, under the conditions prevailing in the work of Franklin and at higher temperatures, was readily confirmed by simple preliminary experiments. Liquid ammonia was condensed upon samples of sodium nitrate and sodium amide weighing 0.5 g. each, in a glass tube which was then sealed, and heated to 60° in a water-bath for three hours. Small amounts of sodium trinitride were identified in the reaction products.

The sodium amide used in the preliminary work was supplied by an American firm which manufactured this material on a large scale for its own use. In a series of experiments samples of this substance weighing 1 g. each were heated to the temperature of fusion in a U-tube through which was passed a slow stream of dry ammonia gas. Small amounts of pure, dry, pulverized sodium nitrate were from time to time cautiously introduced through a side arm into the fused amide. As the reagents came into contact, a reaction of almost explosive violence ensued, accompanied by occasional flashes of light, and a white vapor was ejected from the tube.

After the addition of several portions of sodium nitrate had been effected, the U-tube was allowed to cool, and water was added drop by drop to the contents until the excess of sodium amide had undergone hydrolysis. The resulting alkaline solution was carefully neutralized with sulfuric acid and was then subjected to the usual tests for the trinitride ion.

Treated with a solution of ferric chloride it showed the deep red coloration indicating the presence of trinitrides in considerable quantity. With silver nitrate solution a pale yellow precipitate, of which silver trinitride was obviously not the sole constituent, was obtained. Under the microscope, portions of the precipitate, after recrystallization on the slide from ammonium hydroxide solution, were observed to consist of a mixture of long needles with strong double refraction, and small spherulites of irregular outline and yellow-to-brown color.

Partial separation of the two substances was effected by treating portions of the original precipitate on the filter with a solution of ammonium hydroxide. The acicular crystals dissolved, leaving a yellow residue on the filter. Careful neutralization of the ammoniacal filtrate resulted in reprecipitation of the white needles, which were identified as silver trinitride by the following qualitative tests: (1) the dry crystals detonated sharply when heated on an iron plate over a Bunsen burner; (2) a filter paper containing some of the crystals was colored deep red by a solution of ferric chloride; (3) the crystals dissolved in dil. nitric acid, yielding the characteristic odor of hydronitric acid; (4) when compared with crystals of silver trinitride under the microscope the substance under investigation showed properties identical with those of the known sample; (5) addition of the sodium salt to a solution containing potassium thiocyanate and iodine resulted in immediate liberation of gas and reduction of the iodine.

The yellow substance remaining on the filter was at first thought to be silver hyponitrite and was considered to be a product of the reaction. Subsequent investigation, however, showed that it was the silver derivative of cyanamide, and that the sodium salt was present in the original amide used.

In further preliminary experiments, 2 to 5 g. of sodium amide was cautiously heated in a small nickel crucible and from 1 to 2 g. of powdered sodium nitrate was gradually introduced into the fused mass. A violent reaction ensued, accompanied by the liberation of ammonia, the emission of sparks, and in some cases, the incandescence of portions of the mass. This last phenomenon appeared to initiate a second stage of the reaction during which the products first formed were decomposed. Hydrolysis of the reaction mixture, and subsequent gravimetric determination of the trinitride formed led, in two typical experiments, to yields of sodium trinitride amounting to 16.5 and 15.6% of the amount called for by the equation

$$NaNO_3 + 3NaNH_2 = NaN_3 + 3NaOH + NH_3 \qquad (3)$$

Some improvement was effected by use of a protective layer of molten paraffin and by mechanical stirring of the reacting mass, resulting in a yield of about 20%.

In the thought that a reduction of the nitrate to nitrite might occur as the first stage of the reaction, a brief study of the interaction of sodium amide and sodium nitrite was undertaken. Addition of small amounts of the nitrite to the fused amide resulted in immediate evolution of gas, followed in every case a few seconds later by a violent detonation. No trace of a trinitride could be detected in the product.

Since the examination of various samples of commercial sodium amide, both domestic and foreign, showed the presence of certain impurities it was found necessary to prepare the amide just before use. This was accomplished by passing dry ammonia over sodium cut from the interior of paraffined sticks of the metal, and heated in a nickel capsule held in the bottom of a large Pyrex test-tube fitted with a 4-hole rubber stopper. Two of the openings served for adit and exit of ammonia. The third accommodated a thermometer, and the fourth permitted the introduction of weighed quantities of sodium nitrate.

During the process of amide formation the blue solutions described by Titherley,[26] but not seen by McGee,[27] were invariably observed. After the reaction had gone to completion, the passage of ammonia was continued until displacement of the resulting hydrogen had been effected, as indicated by the complete absorbability of samples of the gas in dil. sulfuric acid. The sodium nitrate, of which a weighed sample was placed in a small bent glass tube set in position in the rubber stopper at the beginning of each experiment, was slowly introduced into the molten amide by gently tapping the tube.

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[26] Titherley, *J. Chem. Soc.*, **65**, 504 (1894).

[27] McGee, THIS JOURNAL, **43**, 586 (1921).

The gas evolved from the reacting mass was quantitatively swept by a current of ammonia into a Schiff nitrometer containing dil. sulfuric acid. The samples of unabsorbable gas thus collected were found to consist of nitrogen and hydrogen. Oxygen and nitrous oxide, as well as the higher oxides of nitrogen, were demonstrated by careful tests to be absent.

The solid products of the reaction were subjected to hydrolysis, and the amounts of trinitride formed and nitrate remaining undecomposed were determined in aliquot portions of the resulting solution. The trinitride was determined gravimetrically as the silver salt, while the nitrate was evaluated by reduction with titanous chloride[28] after removal of the trinitride by treatment with a saturated solution of silver sulfate.

The data obtained in six experiments are recorded in Table I.

TABLE I

SODIUM TRINITRIDE FROM SODIUM NITRATE

| Expts. | $NaNO_3$ used in reaction, g. | $AgN_3$ obtained, g. | Yield of $NaN_3$, % | Nitrogen evolved Cc. (corr.) |
|---|---|---|---|---|
| 1 | 0.0900 | 0.0959 | 60.5 | 13.63 |
| 2 | .0814 | .0869 | 60.6 | 11.66 |
| 3 | .0890 | .0899 | 57.3 | 11.42 |
| 4 | .0894 | .1019 | 64.7 | 13.04 |
| 5 | .0780 | .0744 | 54.1 | 14.74 |
| 6 | .0776 | .0899 | 65.7 | 10.95 |

The weights of sodium nitrate listed in the second column of Table I were calculated by subtracting from 0.1000 g., the amount initially taken in each experiment, the amounts of nitrate found by analysis to have remained undecomposed in the apparatus. The fact that appreciable amounts of the nitrate escaped ammonolysis is attributable to the almost explosive character of the reaction, and the consequent spattering of small portions of the reacting mass upon the cooler upper portions of the vessel.

The percentage yields of sodium trinitride listed in the fourth column were calculated on the basis of Equation 3, in accordance with which one molecule of sodium nitrate should yield one of the trinitride. That higher yields were not obtained in the present experiments is probably ascribable to a secondary reaction involving thermal decomposition of a part of the trinitride, with formation of sodium and nitrogen gas, as expressed by the equation

$$2NaN_3 = 2Na + 3N_2 \qquad (4)$$

In an atmosphere of ammonia gas the metallic sodium would obviously tend to react, forming sodium amide and liberating hydrogen:[29]

$$2Na + 2NH_3 = 2NaNH_2 + H_2 \qquad (5)$$

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[28] Knecht, *J. Soc. Chem. Ind.*, **34,** 126 (1915).

[29] Control experiments, in which mixtures of sodium amide and sodium trinitride were heated in an atmosphere of ammonia to a temperature of 230°, resulted in the liberation of appreciable amounts of nitrogen and hydrogen.

Experimental confirmation of these conclusions is afforded by the appearance of nitrogen and hydrogen gases as products of the reaction.

The total amount of sodium nitrate introduced in each case has been accounted for by ascertaining (a) the quantity of this substance remaining undecomposed at the end of the experiment, (b) the amount of nitrate equivalent to the sodium trinitride formed (Equation 3), and (c) the amount equivalent to the volume of nitrogen liberated during the experiment (Equation 4). The sum of these three quantities was found in each of the six experiments to approximate satisfactorily the amount of sodium nitrate (0.1000 g.) originally taken. The data are recorded in Table II.

TABLE II

DISPOSAL OF SODIUM NITRATE DURING REACTION WITH SODIUM AMIDE

| | Grams of sodium nitrate | | | |
|---|---|---|---|---|
| | A | B | C | |
| Expts. | Undecomposed | Converted to $NaN_3$ | Converted and then decomposed | Total A + B + C |
| 1 | 0.0100 | 0.0544 | 0.0345 | 0.0989 |
| 2 | .0186 | .0493 | .0295 | .0974 |
| 3 | .0110 | .0510 | .0289 | .0909 |
| 4 | .0106 | .0578 | .0330 | .1014 |
| 5 | .0220 | .0422 | .0373 | .1015 |
| 6 | .0224 | .0510 | .0277 | .1011 |

In Expt. 5 the prevailing temperature was 210°, and in all others, 175°. The average total quantity of sodium nitrate accounted for amounts to 98.6% of that taken at the outset. This appears to offer satisfactory confirmation of the theory presented.

The highest yield of sodium trinitride yet obtained amounts to 0.657 mol. of sodium trinitride per mol. of sodium nitrate, or to 65.7% of the amount required by Equation 3. It seems probable, however, that (1) by careful regulation of the temperature of the reaction, (2) by "toning down" its explosive character through addition of soluble or insoluble inert material to the reacting mass, and (3) by suitable modification of the method of bringing the reagents together, the thermal decomposition of the trinitride may be minimized, with the result that yields approaching the calculated may be obtained.

## Summary

Evidence is submitted in support of the contention that hydronitric acid may be regarded not only as ammonitric acid, but also as *hydrazonitrous* or as *ammono-hydrazonitrous acid.*

From this viewpoint all methods that have hitherto been used for the formation or preparation of hydronitric acid may be considered to involve initial solvolysis of an oxyacid of nitrogen or one of its derivatives, with at least incipient formation of a normal acid belonging to the ammonia series, to the hydrazine series, or to both, and ultimate desolvation of this acid.

Sodium nitrate has been found to undergo ammonolysis in contact with fused sodium amide, with formation of sodium trinitride. At 175° a yield as high as 65.7% has been obtained on the basis of the equation, $NaNO_3 + 3NaNH_2 = NaN_3 + 3NaOH + NH_3$.

Higher temperatures favor thermal decomposition of the sodium trinitride, which is probably formed at first in theoretical quantities.

ITHACA, NEW YORK

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[CONTRIBUTION FROM THE PHYSICAL CHEMISTRY LABORATORY, UNIVERSITY OF BRISTOL]

# A NEW SORPTION BALANCE[1]

BY J. W. McBAIN AND A. M. BAKR

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In a paper by the authors[2] on sorption by charcoal it was mentioned in a footnote[3] that a new and improved method had been worked out, which was capable of being applied to all measurements of sorption by solid surfaces. This paper will give the details of the construction and use of the "Sorption Balance."

The earlier method had necessitated the experiment being concluded whenever a reading was desired, so that it was not possible to observe the behavior of the same specimen of charcoal under different conditions of sorption, and it was consequently necessary to assume that different specimens of the same preparation of charcoal exhibited exactly the same properties. Though this was the case in the majority of the experiments made, it is clear that through accidental treatment the activity of the specimen might be very appreciably altered, so that it would be much more satisfactory if a whole isotherm or a complete series of isotherms could be obtained for the same specimen of sorbing material, which was not exposed to any foreign gas or vapor between the experiments.

Many attempts were made by the authors to devise an apparatus embodying these desirable features but, as all the earlier attempts with metal springs and balances of various designs failed owing to the properties of the spring undergoing permanent alteration under the temperature changes that were necessary, only the final form of the apparatus will be described.

The essential part of the apparatus consists of a spring balance, the spring being made of a fine silica fiber. The preparation of the balance will first be briefly described. Long, thin fibers of silica, about 75 to 150 cm.

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[1] The manuscript of this paper was posted by registered post on April 7, 1925, but was lost in transit (compare *Nature*, July 4, 1925, p. 14, vol. 116); it is now submitted unaltered as other laboratories have been in the meantime making extensive use of our balance as here described.

[2] Bakr and McBain, THIS JOURNAL, **46**, 2718 (1924).

[3] Ref. 1, p. 2722.