

test tube is thus avoided and the extract is more easily dried in the small vessel.

This construction of the apparatus is compact, occupying but a small space; is easily manipulated, requiring but little attention, and is economical in respect of the use of gas, a single lamp being sufficient to operate a bath containing four tubes. It has the additional advantage that all parts are open to inspection so that the progress of the extraction can be watched at all times if necessary. It also has the advantage that both the extract and the residue can be weighed and thus a check on the results be obtained. The volatilization of the solvent should be so regulated as not to accumulate any liquid in the extracting tube.

ABSOLUTE ALCOHOL.

SECOND PAPER.

BY EDWARD R. SQUIRE, M. D., OF BROOKLYN, N. Y.

Read before the New York Section, June 2, 1892.

A VERY considerable experience in the manufacture of so-called "absolute alcohol" for the market up to 1884 convinced the writer that really anhydrous alcohol had not yet been obtained.

During the latter part of 1883 and the early part of 1884 the subject was investigated with care, and the results were published by the writer in the *Ephemeris* for May, 1884, 2, 522. This paper opens with the following paragraphs, which are as true to-day as they were ten years ago:

"It appears to be very certain that no alcohol has as yet been rendered entirely anhydrous, and therefore the term 'absolute,' as applied to any yet made, is not strictly correct. For all practical purposes, however, it is a very convenient term by which to designate a rather indefinite substance, but one now applied to a great many important uses, and therefore itself growing in importance.

"It is not difficult to get alcohol practically free from all impurities, including water as one, but to free it from the last one-thousandth part of water is very difficult indeed—so difficult, that traces of this ultimate fraction of water have, so far, always been retained. Hence in considering it as absolute alcohol, it can only be regarded as being freer from all other impurities than from water, and as being more or less free from

water. No alcohol should be called absolute, however, that contains less than 99.4 per cent. by the best determinations. When carefully freed from all impurities except water, specific gravity becomes a very accurate indication of strength, and it has always been relied upon as the decisive test of strength. For many years each careful observer reduced the specific gravity little by little to those specific gravities upon which the tables of the present day are based, by using new appliances which the progress of knowledge supplied, and the object of this note is to show that improvement in these appliances is not yet at an end."

The paper then goes on to show that by slow, cold percolation through quick-lime and simple distillation, alcohol in large quantities had been obtained that was of lower specific gravity than the lowest given by the best of the authorities on the subject. Over thirty determinations were found, and hardly any two of these were in fair accord. Some of the best and most generally quoted of these authorities are examined and discussed with the conclusion that it is particularly unfortunate that the three most generally accepted authorities should, by the construction of their elaborate tables upon erroneous bases, have misled the world for so many years upon this enormous alcohol interest.

Then, commencing with an alcohol that was already below all the authorities found, and yet known to contain traces of water, the attempt was made to reach a still lower degree of hydration. The means adopted, and the checks upon the work, and the results are carefully described in detail with the hope and expectation that more capable hands would go over the work to correct or confirm it, since it was much too important to be accepted without confirmation by other investigators. Nearly nine years have now passed since the publication of this paper, and yet notwithstanding the importance of the subject, no further investigations have been noticed.

The specific gravities previously reached were, at $\frac{15.6}{15.6}$ C., 0.793811 (Drinkwater);¹ $\frac{15.6}{15.6}$ C., 0.7938 (Fownes);² $\frac{15.6}{15.6}$ C., 0.7939 (Tralles);³ $\frac{15.6}{4}$ C., 0.79367 (Mendelejeff).⁴ This last specific gravity when brought to the terms of the others, namely $\frac{15.6}{15.6}$ C., is 0.79391.

¹ Quoted from *Memoirs of the Chemical Society*, 3, 450.

² Quoted from *Fownes' Manual of Chemistry*, third edition, 591.

³ Quoted from *Watt's Dictionary of Chemistry*, 1838, 1, 95.

⁴ Quoted from Roscoe and Schorlemmer, *Treatise on Chemistry*, 3, Part 1, 299.

The specific gravity reached at that investigation was, at $4\frac{1}{2}$ C., 0.802566; at $15\frac{1}{4}$ C., 0.793260; at $15\frac{5}{8}$ C., 0.79279; at $25\frac{1}{4}$ C., 0.78496; at $25\frac{5}{8}$ C., 0.79350; at $25\frac{5}{8}$ C., 0.78573. Thus the reduction of specific gravity effected by the work of that paper was from about 0.79380 to 0.79350 at the temperature of 15.6 C. (60° F.) compared with water at the same temperature of 15.6 (60° F.), or 0.00030, which in strength is equivalent to nearly one-tenth of one per cent.—0.000338 being the difference in specific gravity for 0.10 per cent.

Judging from the circumstances attending the percolations and distillations and from the want of constancy in the boiling point as shown by slight differences in specific gravity of different fractions of distillate, the writer was convinced that this alcohol was not quite anhydrous, and supposed that by modification of process and by better management a specific gravity of about 0.79340 should be attained. The further experience of the past nine years has rather tended to confirm that judgment, and therefore it was concluded to return to the subject at this time, and to review the former work with different methods and some modifications of process, and some improvements in apparatus suggested by longer experience.

APPARATUS OF PRECISION FOR THE SPECIFIC GRAVITY OF LIQUIDS.

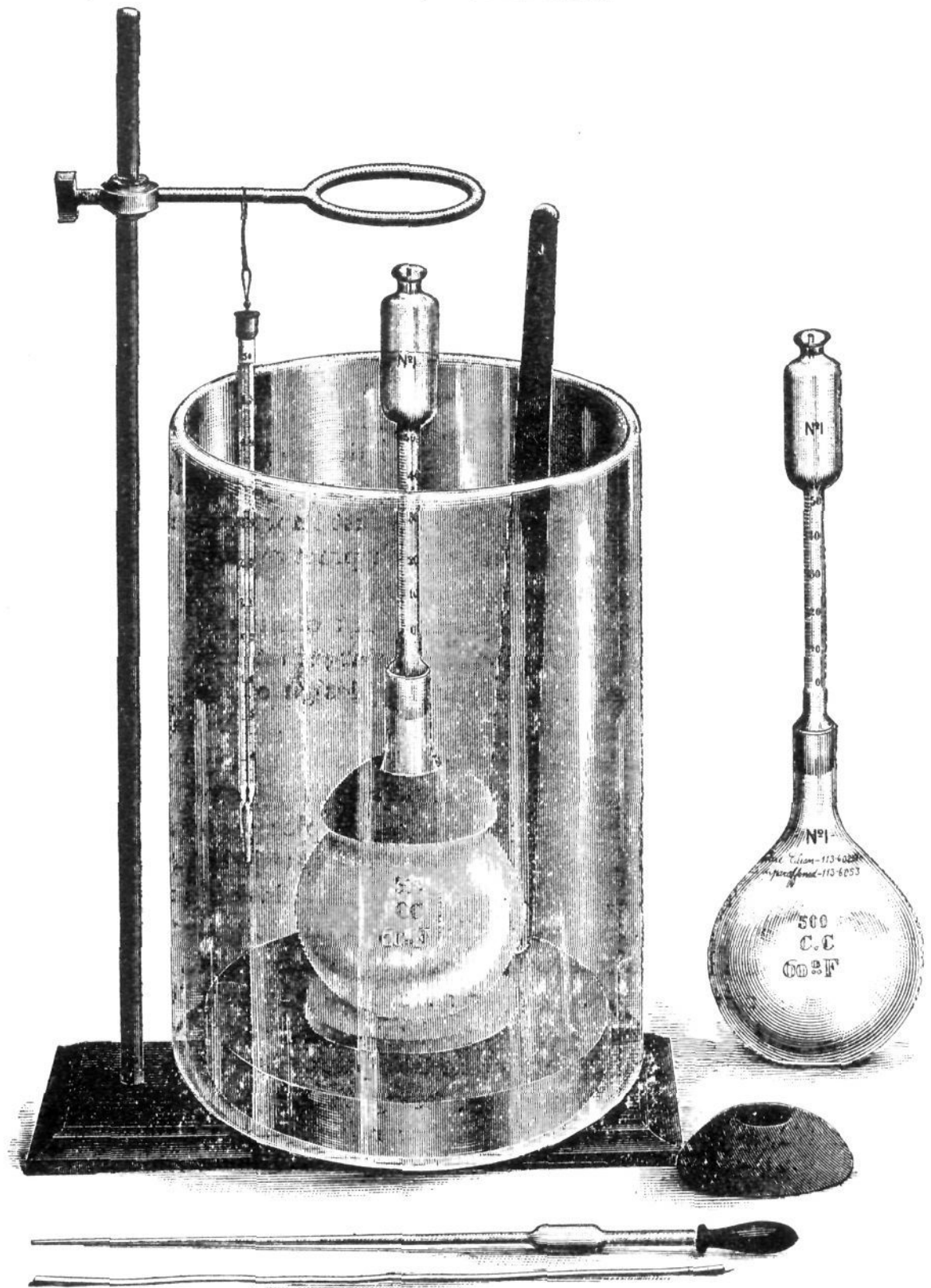
As specific gravity is the sole indication of strength to be relied on, and as alcohol changes very much in volume by changes in temperature, accurate and highly sensitive apparatus was of the first importance in the determination, and it was thought that improvements could be made in the apparatus formerly used.

A standard thermometer was used, made by Hicks of London from Powell glass, standardized and graduated at Kew in 1884, about eight years after it was made. This instrument had its present error determined by comparison with the international standards at the U. S. Bureau of Weights and Measures in Washington through the kindness of the superintendent, Prof. T. C. Mendenhall, and its indications of temperature were read to 0.1° C.

Several very accurate specific gravity bottles of 100 and 500

grams were used, but the one relied upon for the final determinations, and shown in the accompanying cut, is supposed to be an improvement upon any hitherto described. The device of constructing a specific gravity bottle upon the design of a thermometer by having a long tube stopper was carried out for the writer many years ago by an expert Paris glass blower, but it had no provision for expansion of the liquids without loss, and was too tall for any ordinary balance. The design was improved and carried out better by the writer in 1884 (see *Ephemeris*, **2**, 529), and was still further improved in 1889 (see *Ephemeris*, **3**, 1162-1166). There was, however, still a recognized defect, in want of room for the expansion of liquids in warming up to room temperatures before weighing, and in so limiting loss by evaporation during expansion and weighing, that a practically constant weight could be attained. This last improvement seems to fairly satisfy these conditions, and in practice answers very well.

The bottle is an ordinary 500 cc. flask old enough to have reached its maximum contraction, and so large that errors of reading and weighing are well within the design of reaching the sixth decimal place with tolerable accuracy. A flask was selected with narrow neck, and the graduation mark low in the neck. The neck was cut off just above the mark and was thickened and shaped for stoppering. A piece of barometer tubing fifteen to twenty cm. in length was expanded into a long stopper at one end and carefully ground into the flask neck. Then 500 grams of recently boiled distilled water was put into the flask and stopper and the whole cooled in an iced bath to 4°C . (39.2°F .). The water in the tube stopper should then be near the lower end, but if not, the end is ground off until the meniscus for 4°C . is just above the ground part of the stopper. The 500 grams of water and the 4°C . temperature being then again accurately adjusted, a mark was made at the lower limb of the meniscus. The bath was then warmed up to 25°C . (77°F .) and when the water had ceased to rise in the tube after the temperature of the bath had been held constant for half an hour, another mark was made, the tube being of such a caliber that the two marks should be about ten cm. (four in.) apart. Any excess in the length of



APPARATUS FOR SPECIFIC GRAVITIES OF LIQUIDS.

the tube above the upper mark was then cut off, and a liberal section of a very much larger tube was put on. This larger portion was about 2.4 cm. (one in.) in diameter, and about seven cm. (2.75 in.) long, drawn into a narrow neck at the top, and the neck fitted with a flat ground glass stopper which had a capillary passage through its center. The tube portion of the stopper was then graduated in fifty equal subdivisions, the scale beginning with 0 at the 4° C. point, and extending to fifty at the 25° C. point. These subdivisions are easily and accurately read to half divisions so that the scale is read to hundredths. Then upon accurate readjustment it was found that the reading with recently boiled distilled water was one—that is $\frac{1}{100}$ of the scale from 4° C. to 25° C. The reading at 10° C. was six, or $\frac{6}{100}$ of the scale. At 15° C. the reading was twenty-five. At 15.6° C. (60° F.) it was twenty-eight. At 20° C. it was fifty-seven, and at 25° C. (77° F.) it was 101. These readings illustrate in a very interesting way the rapidly increasing ratio of expansion for water. Without correction for expansion of the flask the increase in volume for 25° C. (4° to 25°) is just 100 divisions of scale, giving a mean value of 4.76 ÷ divisions to each 1° C. But the expansion for the first six degrees (4° to 10° C.) is only six divisions of scale, or one division to each 1° C. while the expansion for the last five degrees (20° to 25° C.) is forty-four divisions, or 8.8 divisions to each 1° C. This scale adapts the flask to taking specific gravities at all the ordinary standards of temperature, and adapts it to correction by readjustment at any time in case of contraction of the glass by age. It also enables the observer to watch the changes in height of the column of liquid and to know accurately when it ceases to rise or fall. When accurately adjusted the weights were taken on a fine balance to 0.0001 gram when chemically clean, and also when the stopper was lightly lubricated with soft paraffine,—these tares being etched upon the bottle, and all the readings of the standard water being entered in the laboratory note book.

BOTTLE IN USE.

The bottle, properly filled, and loaded with a leaden collar to steady it, was always used in a glass water-bath, the water of the bath being kept above the level of the reading lines, and

these read through the glass and water. The thermometer was suspended in the bath with the bulb opposite to the upper part of the bottle, and was also read through the glass and water, and a wooden stirrer was used to keep the water uniform throughout. When the temperature of the bath had been held steady to the required temperature for ten minutes after the column in the tube had ceased to rise or fall, the adjustment to the required graduation mark was made by passing the long capillary pipette and the very narrow strip of blotting card paper, shown in the cut, down through the tubulure in the stopper. In order to know how nearly the temperature of the liquid in the bottle agreed with that of the water of the bath, a pair of compared thermometers were used, and it was found that with either water or alcohol in the bottle, the temperatures were in accord to about 0.05°C . after all perceptible change in the column had ceased, and the bath had been held constant for five minutes; and upon this experience ten minutes was adopted as the uniform time of waiting before the final adjustment. Then it was found that no constant weight of bottle and contents could be obtained so long as these differed more than one or two degrees from the room temperature of scale-case and weights, and therefore a second bath was used after adjustment to bring them accurately to room temperature, and then they were allowed to stand on the balance with the case closed until they reached a fairly constant weight. As all this management required time, it became necessary to know whether there was any material loss in weight between the time of adjustment and the final weighing, and on trial it was found that with water the bottle and contents, either in the bath or out, did not alter perceptibly in weight within forty-eight hours, and with the alcohol under the same conditions, the loss in a warm room was not greater than 0.04 gram in forty-eight hours, or less than one milligram per hour. This loss was therefore neglected as being well within the sphere of unavoidable errors.

The weighing was done upon a large balance sensitive to 0.002 gram, and this was readjusted for each accepted critical weighing. The weights used were of brass, of a specific gravity of about 8.383, with platinum fractions of a gram. They were not stand-

ardized, but as they agreed among themselves, and as all the weighings were made with the one set, this gave no source of error. The critical weighings were made with the barometer not far from 760 mm. (thirty inches), and no correction for barometer was attempted. All weighings were made against water at the same temperature taken as unity, and therefore corrections for expansion of glass were avoided. Neither were any corrections attempted for the use of brass weights, because such corrections take the results out of the range of ordinary daily practice and tend to confusion rather than to practical utility. As the weighing was done to 0.002 gram in a 500-gram bottle the results are stated to the sixth decimal place, and although the sixth decimal cannot be trustworthy, even to twice the indicated value, it is still believed to be of more value than if expressed in the nearest unit of the fifth place. In view of the rapidly increasing importance of specific gravities of liquids, it is very desirable to give a more definite value to this sixth decimal place. The writer has 100-gram specific gravity bottles of the same construction which can be easily weighed on a fine balance to 0.0001 gram, but as yet the measuring or volume adjustment cannot be brought up to this high degree of precision.

LIME PERCOLATOR.

It was confidently expected that a more perfect dehydration might be obtained by often repeated percolation through lime than by the former process of prolonged shaking with lime, provided the percolation could be done without air contact, as was not done in the processes of the former paper.

A very large calcium chloride jar, holding almost a kilogram of lime, with large chamber below, was nearly filled with strata of granulated quick-lime alternately fine and coarse, the whole supported at the contraction below on a layer of glass wool. At the top a rubber stopper with two perforations was used, supplied with two glass tubes bent twice at right angles, the short leg projecting just through the stopper, while the long leg extended downward outside the jar. The tubulure of the chamber below was fitted with a rubber stopper of one perforation supplied with a glass tube, bent downward within so as to draw all the liquid

out, and bent upward without to receive rubber tubing. The object was to deliver the alcohol on top of the lime, draw it through the lime by means of the Sprengel water-pump, and then draw it out of the chamber below the lime to be again passed on top, and again through the lime, and this without contact with any air that had not been thoroughly dried. Near the lime percolator on each side was placed a bottle of two liters' capacity, each fitted with rubber stoppers supplied with a long and a short tube, the short tube passing just through the stopper, and the long one going to the bottom and so bent that its end reached the very lowest point in the bottle. A little farther off from the percolator on one hand was a large apparatus for drying the inspired air, and on the other hand the Sprengel water-pump. In the drying apparatus the air entered through a tube filled with caustic baryta, then bubbled through concentrated sulphuric acid and then ascended through a large jar of calcium chloride to the outlet. By means of rubber tubing and glass **T** and **Y** tubes these various parts were so connected that by means of screw pinch-cocks some passages could be stopped and others opened to the effect that the alcohol was easily and conveniently drawn from one bottle upon and through the lime into the other bottle, well dried air taking its place, until the bottle was empty and its fellow on the other side full. Then the closed pinch-cocks were opened, and the open ones closed and the alcohol was drawn back through the percolator to the other bottle, and so on. With a partial vacuum of only about 100 mm. (four inches) about four of these percolations were made in a day, and generally the alcohol was passed through about twenty-five times before being distilled.

APPARATUS FOR THE DISTILLATIONS.

The distillations were all made in one arrangement of apparatus. A round bottomed flask of about two liters' distilling capacity was held on a round bottomed water bath over a gas flame. The flask was fitted with a good cork that had been well dried by immersion in hot paraffine. This cork was perforated for a small tube through which to aspirate dry air, or the charges of alcohol, and also for the large end tube of a Hempel tube

about thirty-five cm. (13.7 inches) in total height, the passage of this tube through the cork being made air-tight by means of paraffine. The Hempel tube was then three-fourths filled with small glass marbles, and from the end of this tube, in the flask, a thermometer was hung. In the bottom of the flask, to prevent explosive boiling, was put a liberal supply of recently ignited fragments of clay, tobacco pipes, and platinum scraps. From the top of the Hempel tube a vapor tube of liberal size, bent twice at right angles, led to the condenser. The condensing tube was of thin glass, about seventy cm. (27.8 inches) long, turned up at one end to receive the vapor tube from the Hempel and bifurcated at the other end, each branch having a well ground glass stop-cock, well lubricated with soft paraffine—the ends of the branches beyond the stop-cocks being contracted and turned down. About fifty cm. (19.75 inches) of this tube passed water-tight through a tinned iron trough capable of holding a good supply of broken ice and water. The two terminal ends of the condenser were connected by rubber tubing with short straight pieces of glass tubing which passed through one hole of two two-hole rubber stoppers. The second hole of each stopper was fitted with a bent glass tube, and these were connected by rubber tubing with the aspiration tube of a competent Sprengel water-pump. The two rubber stoppers, through which the small glass tubes passed, fitted all the specific gravity bottles, and the arrangement admitted of distilling into one or the other bottle at will, and of changing bottles without the contact of much air, without interruption and without breaking the vacuum. The distillate was usually divided into seven fractions for each distillation. The first two in 100-gram specific gravity bottles, the third in the 500-gram specific gravity bottles, the fourth, sixth and seventh in 100-gram bottles, and the fifth in a 500-gram bottle.

In making the distillations the boiling was commenced under a partial vacuum of about 300 mm. (twelve inches), and without condensation in order that the pump might draw off all dissolved air and gases, and any liquids of low boiling point. Under this diminished pressure the boiling always began at about 50° C. (122° F.), and the temperature rose steadily to

about 67° C. (152.6° F.) before any considerable vapors went beyond the Hempel tube. Then the ice and water were put in the condenser trough, and the first 100-gram specific gravity bottle was filled under this partial vacuum. Then the vacuum was allowed to run down to about twenty-five mm. (one inch) and the remainder of the distillation was conducted under that slight minus pressure.

THE ORIGINAL ALCOHOL.

The following trials were all made with an alcohol that was always at hand in great abundance. It was believed to be quite free from all impurities excepting water and to contain only about 0.1 per cent. of water. The specific gravities taken with much care were as follows: At $\frac{4}{5}$ C., 0.802818; at $\frac{10}{15}$ C., 0.798018; at $\frac{15}{15}$ C., 0.794258; $\frac{15.6}{15.6}$ C., 0.793824; at $\frac{20}{20}$ C., 0.790714; at $\frac{25}{25}$ C., 0.787332. Provided the specific gravities given by Drinkwater, Fownes, Tralles and Mendelejeff were taken with as much accuracy as these now given here, this is the alcohol that, up to the date of these investigations, has been considered to be anhydrous, and it is the basis of all the best tables.

VALUE OF SPECIFIC GRAVITY IN PERCENTAGE.

Early in the investigation at this time, it became desirable to know with more accuracy than heretofore, the percentage value of specific gravities where there is so very little water present. To determine this point a portion of the original alcohol above described was used, and 1345.174 grams was carefully weighed off. To this portion 2.696 grams of distilled water was added giving a dilution of almost exactly 0.2 per cent. and the mixture was allowed to stand twenty-four hours, when the specific gravities were taken.

Specific gravity	At $\frac{4}{5}$ C.	At $\frac{10}{15}$ C.	At $\frac{15}{15}$ C.	At $\frac{15.6}{15.6}$ C.	At $\frac{20}{20}$ C.	At $\frac{25}{25}$ C.
Original alcohol	0.802818	0.798018	0.794258	0.793824	0.790714	0.787332
0.2 per cent. dilution	0.803434	0.798662	0.794932	0.794500	0.791362	0.787938
Difference for 0.2 per cent.	0.000616	0.000644	0.000674	0.000676	0.000648	0.000606
" " 0.1 " "	0.000308	0.000322	0.000337	0.000338	0.000324	0.000303

If these differences were interpolated and plotted they would give a form of curve for alcohol not hitherto known to this writer.

Next this portion of diluted alcohol was passed through the

lime percolator twenty-five times in six days to ascertain what proportion of this water might be abstracted by this treatment, and how near to a constant specific gravity or boiling point such alcohol would be. It had been recognized in the former investigations that a thermometer in the boiling alcohol was not an accurate indicator of the boiling point. In a clean glass flask the boiling was so nearly explosive that differences of a degree were often noticed within a very few seconds. Even with fragments of clay tobacco pipes and platinum scraps in the vessel the thermometer would rise and fall enough from time to time to preclude accurate observation of the constancy of the boiling point. The specific gravities of the fractions of distillate were, however, much better indications of the boiling point, since if these were constant, the boiling point would necessarily be also constant, and until these fractions were constant in specific gravity they could not be uniform in boiling point, and as long as they were not constant they could not consist of alcohol alone. That is, any mixture of two liquids as different in boiling points as alcohol and water could not distill at a constant boiling point, nor yield a distillate of constant specific gravity; and now it is known that a tenth of one per cent. of water in alcohol gives a very decided difference in specific gravity. The specific gravities given by the seven fractions of distillate from the portion of diluted alcohol that had been passed twenty-five times through the lime percolator were as follows, in the order in which they were received, all taken at $\frac{15.60}{15.60}$ C.: First, 0.79414; second, 0.79403; third, 0.79402; fourth, 0.79400; fifth, 0.79392; sixth, 0.79400; seventh, 0.79404. Then all the fractions were put together and the specific gravity of the mixture was 0.793954.

These results are accepted as proving conclusively that no part of the distillate was anhydrous alcohol, and that about 0.16 per cent. of the water added was taken out by the lime, leaving about 0.04 per cent. of the water put in that was not taken out by the lime.

DEHYDRATION BY METALLIC SODIUM.

Many times in the literature of late years metallic sodium has been recommended for the detection of water in alcohol, and for depriving alcohol of the last traces of water. But such recom-

mendations were not easily understood in view of the fact that at ordinary temperatures sodium decomposes alcohol rather rapidly. It might, however, also abstract water. Then, too, the well-known influence of cold in modifying chemical action might be available, so it was concluded to try the effects of sodium at low temperatures. About two liters of the alcohol was well cooled in a bath of snow and salt to about 17° C. (1.4° F.) and was held at that temperature or lower for about forty-eight hours. Throughout the day time of this period small pieces of sodium, two or three grams at a time, were dropped into the alcohol with the least practicable admission of air, and this was repeated five or six times a day, so that an aggregate of about forty grams of sodium was used. The sodium, instead of floating, went at once to the bottom, and the reaction was very moderate. This alcohol was then carefully distilled off from the sodium ethylate, the distillate being received in fractions, which varied considerably in specific gravity. The lowest fraction was, however, at $\frac{15.6^{\circ}}{15.6^{\circ}}$ C., 0.793713, and the average of the fractions was not below the 0.793824 of the original alcohol. This negative result is given because it may possibly save others from going over this ground.

Experiment 1.—A portion of good quick-lime was slaked with just enough water to make a fine dry powder. This was put into the center portion of a piece of three-inch wrought iron pipe, the two end portions being filled with quick-lime. Caps being screwed loosely on the ends the pipe was subjected to a dull red heat during four hours, and then cooled over night. About 500 grams of this lime was put into a bottle of 4.25 liters' capacity, and, 2.5 liters of the original alcohol having been poured upon it, the bottle was shaken half an hour at a time, in a mechanical shaker, during an aggregate of sixty hours in five weeks. The alcohol was then filtered off from all traces of lime, with as little air contact as practicable, and was aspirated into the distilling flask and distilled. The specific gravities of the seven fractions taken at $\frac{15.6^{\circ}}{15.6^{\circ}}$ C. were: First, 0.793851; second, 0.793839; third, 0.793680; fourth, 0.793701; fifth, 0.793679; sixth, 0.793400; seventh, 0.793701; an eighth fraction was 0.793620.

The sixth fraction of this series was rejected as being too low, and quite out of accord with the other elements of the series, but all the other weighings were trustworthy.

Experiment 2.—The mixed fractions from experiment 1 were passed through the lime percolator about ten times and then again distilled, when the fractions gave the following specific gravities at $\frac{15.6^{\circ}}{15.6^{\circ}}$ C.: First, 0.793960; second, 0.793801; third, 0.793582; fourth, 0.793639; fifth, 0.793606; sixth, 0.793576; seventh, 0.793549; second weighing of the seventh fraction gave 0.793499. At the first weighing it had not reached a constant weight, while at the second weighing, thirty minutes later, there may have been a very slight loss.

Experiment 3.—A fresh portion of the original alcohol was passed twenty-five times through the lime percolator, and distilled with the following results: First, 0.793960; second, 0.793811; third, 0.793639; fourth, 0.793582; fifth, 0.793576; sixth, 0.793561; seventh, 0.793499. This last weighing, although reached here for the second time (see above), must be received with doubt, because it is not in accord with the other elements of its series.

When these results are carefully examined it will be seen that with better apparatus and better management, the results of the former investigations were not quite reached. If 0.000338 be the specific gravity value of 0.1 per cent. in alcoholic strength, then the former results, which reduced the specific gravity from about 0.793824 to 0.793500, or 0.000324, indicated a difference in strength of 0.09585 per cent.

Taking the mean of three trustworthy readings of this paper, namely 0.793549, 0.793576 and 0.793561, which mean is 0.793562, and subtracting it from the original alcohol, 0.793824, the difference is 0.000262 and this is equivalent to 0.07751 per cent.

The writer was unable to carry through the design of this paper for want of time and leaves one or two methods of dehydration that have occurred to him, still untried, and he also very much regrets having to leave, for the present, a thorough repetition of the former successful process of nine years ago. It seems to the writer very certain that up to the present time no really anhydrous alcohol has been obtained, and therefore with the

present better apparatus and management, and especially by prolonged contact with the lime, through the old process, better results might reasonably be expected.

PYROXYLIN, ITS MANUFACTURE AND APPLICATIONS.

BY WALTER D. FIELD.

Received June 17, 1893.

PART I.

BY the term pyroxylin is understood the soluble nitric ethers of cellulose, namely, the di, tri, tetra, and penta-nitrates. From the date of the use of pyroxylin in photography by Scott Archer in 1851, the number of its uses has increased until, at the present time, tons of the lower nitrates of cellulose are produced yearly. With this increase of production improved methods of manufacture have been evolved.

So general has become the use of this material that to-day it is to be found everywhere. In the form of celluloid it is before us constantly. As a varnish it is used on penholders, pencils, silver and brass ware. Articles are bronzed with it as a medium. An artificial leather has been produced with it, many thousands of yards of which have found a ready market. These applications are all made, with the exception of celluloid, by the use of a solution of pyroxylin.

The first part of this paper pertains to the fibers and their selection for the purposes of nitration; the second part to the processes used for nitrating. The third and fourth parts will treat of the various methods of washing and drying the pyroxylin and of its solvents and uses.

Selection of the Fiber.—Cotton fiber, wood fiber, and flax fiber in the form of raw cotton, scoured cotton, paper, and rags are most generally used and give the best results. The fibers differ greatly in their manner of nitrating, a difference due undoubtedly to their physical structure; hence a given fiber demands a method of nitrating suitable for that particular fiber.

The cotton fiber is a flattened hollow ribbon, or collapsed cylindrical tube, twisted a number of times, and closed at one