	SO ₂	O	N by difference.	Original O calculated from N.	O "Absorbed."	O Absorbed SO ₂ .
CROWDER. This J., 1891, 303	4.86 5.12 5.21 5.42	10.08 10.17 10.44 10.01				
Average	5.15	10.33	84.52	22.31	0.83	.33
	6.34 6.43 6.54 6.57	8.82 8.53 8.82 8.47				
"	6.47	8.66	84.87	22.40	7.27	1.12
	7.03 6.83 6.21	8.0 8.08 9.43				
.....	6.60	9.10	84.21	22.23	6.44	0.91
	7.43 7.31 7.06 7.03	7.06 8.06 8.32 8.32				
"	7.21	8.16	81.63	22.34	6.97	0.97
FISCHER. Dingl. J., 258, 27	0.96 1.52 3.81 8.26 7.53	18.4 16.6 12.5 5.9 7.5	80.61 81.88 83.69 85.84 84.97	21.29 21.61 22.07 22.66 22.42	1.93 3.40 5.76 8.50 7.39	2.01 2.30 1.51 1.93 0.98
	2.48 2.62 8.43 4.02 5.80 9.3	14.8 16.0 3.9 10.7 10.6 5.0	82.72 81.38 87.67 84.38 83.60 85.70	21.84 21.49 23.30 22.28 22.08 22.62	4.56 2.87 10.81 6.66 5.68 8.12	1.84 1.1 1.28 1.35 0.98 0.87
SCHURER-KESTNER. Bull. Soc. Chem., 23, 137	6.33 8.57	8.66 6.77	85.01 84.66	22.44 22.29	7.45 7.01	1.16 0.82
L. T. WRIGHT. Private memoranda	6.6 7.2 7.2 7.6 8.4 8.6	8 6.7 7.7 6.0 6.5 5.5	85.4 86.1 85.1 85.5 85.1 85.9	22.54 22.73 22.47 22.57 22.47 22.68	7.07 8.8 7.57 8.07 7.57 8.58	1.20 1.22 1.05 1.06 0.90 0.99
LUNGE'S "Normal" gas	8.59	9.87	—	—	—	0.37

DISCUSSION.

Sir WILLIAM RAMSAY asked whether it was certain that the formula of the oxide of iron produced was Fe₂O₃. The main argument seemed to be conceived on a difference of oxygen, an error in the estimation of which would make that difference greater or smaller.

Mr. J. H. COSTE said that sulphur trioxide was always produced when coal gas was burned without chilling the products.

Sir WILLIAM RAMSAY asked whether it was allowed for.

Mr. J. H. COSTE said that the gases from a calorimeter, like Boys' or Junker's, always showed sulphur dioxide, apparently because the product had been chilled; but if gas were burned in the Referees' apparatus for determining sulphur practically the whole of the sulphur came off as sulphur trioxide. Sulphur dioxide was not usually found.

The CHAIRMAN said that that might account for the expected discrepancy in the analyses in the second column.

Mr. D. M. MACKECHNIE said, with regard to Sir William Ramsay's remarks on the degree of oxidation of the iron present, ferrous oxide was admittedly present and might be there in quantity.

Mr. LEWIS WRIGHT, in reply, said in regard to the matter raised by Sir William Ramsay, the amount of sulphur trioxide formed under these circumstances, would hardly, in his opinion, account for the ratios shown.

Of course some magnetic oxide was formed and that affected the ratio. He did not know if anyone had determined that; but there were other well-known secondary reactions taking place. Some of the claims which had been made to the production of a burner gas containing nearly 18 per cent. of SO₂ in special types of furnaces could hardly be explained in the face of the analyses which came from all kinds of practices, not only from his own but that of many other chemists engaged in the manufacture of sulphuric acid. On the question of the possible analytical error in the absorption of oxygen which was suggested by Dr. Lunge, when they were dealing with gases of such extreme dilution, it was obvious any of the many possible errors in technical gas analysis might happen and would then affect the ratio. If after a burner gas was taken away through various channels, air was allowed to enter, that was to say, if there was a mixture of burner gas and false air, or if a dilute gas was taken off directly from roasting pyrites, the same high ratio was found in each case.

THE HEAT TEST OF EXPLOSIVES.

BY A. O. EGERTON.

It is satisfactory to find that interest has been taken in the paper on the heat test published in this Journal, 1913, 331, Robertson and Smart having criticised several of the deductions therein (see this J., 1913, 967).

The results of certain experiments, described in the fore-mentioned paper, were only explicable, in the opinion of the writer, by considering that an explosive can attain a state of equilibrium within a closed heat test tube. In fact, it was suggested that the dissociation pressure of the explosive compound was measurable, and would act as a good guide to its stability. Such a statement has been considered untenable, because decomposition of an explosive can proceed to completion—it is not a reversible process.

Explosives to which the heat test is applicable, on heating at a constant temperature for a long time, gradually deteriorate and eventually decompose; in fact on heating they are all liable to ignite of their own accord. This could not be so if the products of decomposition were in equilibrium with the explosive; indeed the products of decomposition tend to accelerate the rate of decomposition. What then is the evidence for the assertion made in the author's last paper?

It was found that the heat test time was little altered by the presence of two test papers, or even two drops, although calculation from the formula derived from the assumption of a regular rate of increase of the concentration of the nitrogen peroxide and its removal by absorption in the drop or paper, would require that the heat test time should be considerably greater: the continuous tests show that for quite a long period the heat test times remain practically constant.

These two facts appear only to be explicable on the hypothesis that equilibrium establishes itself within the tube. For how otherwise would the results of a continuous test not give decreasing times, or the presence of two drops instead of a single one not cause an increase in the time of the first test?

It will be well to consider other possible reasons. Firstly the concentration of "reacting" gas may be greater than

tested at long intervals, after the same period of time has elapsed from the commencement, nearly the same heat test times are obtained. There remains Dr. Robertson's suggestion that during a continuous test the nitrogen peroxide is reduced to nitrogen, this action increasing as time goes on much in the same proportion as the nitrogen peroxide is evolved. If this were so, two tubes treated similarly and heated for a certain time, should contain the same amount of nitrogen peroxide; if they were then tested the one with one drop or paper and the other with two, the results should be very different; they are, however, nearly the same. There is also direct evidence that this is not a plausible explanation, as will be shown later: indeed, from the results of the Will test it would be exceedingly unlikely that the reduction would be sufficient to account for the constant heat test results, especially as the temperature at which a heat test is carried out is so much lower than that for the Will test.

Smart has pointed out that oxygen, nitrous and nitric acids, nitrogen trioxide and peroxide, and nitric oxide are all reacting gases so far as the heat test is concerned: if constant continuous test results were only obtained for the potassium iodide paper, it would be easy to understand, for the reaction on the paper is an oxidising action and that on the drop is due to nitrous acid. It appears conceivable that the nitric oxide found by reduction of nitrogen peroxide might act as a carrier of oxygen, being alternately reduced and oxidised, and this might account for the constant results; but as the nitrogen peroxide increased, one would expect the nitric oxide to increase, and the test times to decrease. It does not seem that such considerations throw any light on the matter.

As none of these explanations suffices to explain the experimental results which had been obtained, it was thought advisable to confirm them, and the following table gives the results of some of the further experiments.

TABLE I.
Guncotton "Conditioned."

KI paper. 70° C.				Naphthylamine drop. 75° C.			
1. Single paper 10' interval.	2. Two papers 10' interval.	3. Single papers 30' interval.	Time.	1. Drops exposed successively.	2. Two drops (in cases marked*).	3. Single drops 30' interval.	Time.
24'	22½'	21½'	30'	23'	24'	24'	60'
4'	8'	2'		10'	10'		
3½'	4½'		60'	9½'		8½'	90'
3½'	7½'	3½'		8½'	10½'		
3½'	7'		90'	9½'		8½'	140'
3'	8'	1½'		10½'	11'		
4'	8'		120'	14'		15'	200'
3½'	8'	2'		18'	17'		
3½'	6'		150'	17'		21'	210'
4'	5'	3'		27'	31'		
5'	10½'		180'				240'
3½'	0'	2½'					
4½'			210'				240'
5'	15'	3'					
5'			240'				270'
5'	15'	4'					
8½'			270'				
9'	11'						

expected and the absorption by the drops or paper less; an upper limit for the value $T_0 = K$ for the drops and papers was found (for the drops $K = 150 \times 10^{-6}$), but even this is not great enough to account for the similar results obtained when two drops are exposed; the value for the absorption by the papers was confirmed, and that for the drops was found to be nearly four times as great. This, then, will not account for the facts. Secondly, during a continuous test the drops or papers may absorb the reacting gases as fast as they were formed, or leakage and dilution of the atmosphere within the tube may occur when the drops are changed; this, however, is also disproved, because, if a continuous test is run alongside a similar sample which is kept in a closed tube and only

Thus after two hours in the case of the paper indicator,
When 14 papers have been exposed, heat test time is .. 8'
 $\frac{8}{4}$ " " " " " " " " " " :: 3 $\frac{1}{4}$ '
 4 " " " " " " " " " " :: 3'
while with the drops after 1½ hours

When 8 drops have been exposed, heat test time is ..	11'
9 " " " " " " " " " "	10 1/2'
10 " " " " " " " " " "	8 1/2'

It has been shown in the other papers that the presence of two papers or two drops during the first test should alter the time of the test considerably; but in fact, experiments show that the time of the first test does not alter.

On the other hand, a simple calculation shows that if the rate of evolution of nitrogen peroxide is about $1.0 \times$

10⁻⁶ mgrm. NO₂ per minute per c.c., the amount of nitrogen peroxide in a heat test tube after two hours should be about 1.6×10^{-3} mgrm., a number which would give a very quick heat test time, and which would not be affected appreciably by the exposure of several papers within the tube.

If the amount remained about 5×10^{-4} mgrm., as the continuous tests seem to show, the difference in tests on exposing different numbers of papers should be about that observed; one can quite realise that the approximate equilibrium would hardly establish itself quickly enough to prevent this effect being observed. The drops absorb much more nitrogen peroxide in colouring up to tint, and if it were not that results after the exposure of very few drops are not greatly different from those where many drops have been exposed during the continuous tests, it would be possible to explain the constant results obtained by absorption in the drop, but that possibility is excluded.

One point to be noticed is that the heat test rises after a certain time, and that this rise occurs sooner, the greater the number of drops or papers introduced. The suggestion of Smart that at the commencement of the continuous test the nitrogen peroxide is reduced by foreign substance, more nitrous acid being thereby obtained, might possibly explain this fact; but it is more probable the result of a slower rate of evolution of nitrogen peroxide after the explosive has been heated some time.

Other experiments were made which confirmed the above results. One experiment was carried out with a sample of dry gucotton at 70° C. for 36 hours (using a drop of α -naphthylamine as indicator). The drops were exposed at intervals of an hour. The test times obtained were practically constant, and no gradual increase in concentration in nitrogen peroxide was detectable.

These results confirm those described in another paper, and show that the reacting gases do not increase regularly as time goes on, but that their concentration remains practically constant. Excluding the idea that reduction to NO and nitrogen might account for these constant results, it does not seem possible to explain them otherwise than by assuming that the explosive is behaving like an ordinary dissociable substance in an enclosed space.

How, then, can the two views be brought into line, and how does an explosive behave when kept at a temperature about 70 to 80° C.?

The explosive, if it were an ordinary substance at a constant temperature (e.g., copper nitroxyl), would get into equilibrium with the products of decomposition. There is little doubt that this is the main condition of affairs; at the same time, in order to account for the instability of an explosive and its liability to decompose rapidly, there must be a second process going on. Certain few molecules (especially in the case of explosives of non-homogeneous structure) are decomposed more completely than those which are merely dissociating, and these are decomposed irreversibly with evolution of considerable heat; at certain centres, then, within the explosive this action begins and may spread rapidly and cause spontaneous ignition. The two actions may be looked on as separate; and the results of heating an explosive, as in a continuous test, are those of the two supposed actions. When the explosive is first heated, the explosive may be somewhat more disturbed than after a considerable heating. The explosive would show sudden increases in the concentration of the "reacting" gases; but no marked increase should occur until the explosive has practically reached a condition in which it is likely to explode. These remarks are borne out by the results of continuous tests.

Leakage or absorption by the reagent have been shown not to account for the approximately constant results of a continuous test. As it is not apparently impossible to prove satisfactorily by means of experiments, such as have been described, whether or not reduction of nitrogen peroxide is sufficient to account for the constant results obtained in continuous tests, another method has been devised for this purpose. The method is based on Ramsay's apparatus for measuring small quantities of gases.

The gas burette, A, ends in a fine capillary tube, B. The side tubes communicate through taps with a bulb containing potash, C, a tube of phosphorus with a mercury inlet, D, and a tube, F, containing the gucotton which can be heated at a constant temperature. The upright tube communicating with the mercury reservoir can be closed by a tap so that the mercury can be shut off from the inside space. The bulb, E, contains pure oxygen. The gucotton in F is heated for some definite time in pure oxygen, and in the meantime the burette, etc., are completely evacuated. The tap to the burette is then opened so that the gas passes chiefly into A (the bulb, F, being small); after closing this tap, that to D is opened and the oxygen burnt up by the phosphorus, and the oxides of nitrogen are decomposed giving nitrogen. The result would be a mixture of CO₂, CO, hydrogen, and nitrogen.

The CO₂ can be partially got rid of by opening to the potash bulb. To get rid of hydrogen a little oxygen is let into the burette, A pumped out and sparked. Then it is let back into the apparatus through the inlet to the phosphorus tube, the nitrogen and CO₂ remaining pass into the burette on opening the tap thereto. The CO₂ can be cooled out by a cup of liquid air over the capillary tube and the residue examined by passing the electric discharge through, by connecting to the mercury reservoir and the small platinum wire at the top of the capillary tube. Its volume can also be measured in the capillary tube.

After two hours' heating, if the rate of evolution of N, NO, and NO₂ from the explosive is constant, the volume of nitrogen obtained should be greater than two cubic millimetres for an ordinary gucotton according to the calculation from the formula giving the rate of evolution of nitrogen peroxides. It is possible, however, to detect a very much smaller quantity of nitrogen by this method.

The results of preliminary experiments have shown that a very much smaller quantity (about $\frac{1}{3}$ cubic mm.) than this is obtained and that this quantity does not seem to increase on prolonged heating for several hours. In fact the results so far obtained support the contention that the explosive substance behaves, in part, like an ordinary dissociable substance.

The method is somewhat difficult to work, but if it can be perfected it should be valuable as an absolute method of measuring the stability of an explosive by the analysis of the gases evolved on heating for different periods of time at different temperatures.

Another experiment was made, in which the bulb, E, was filled with pure CO_2 , instead of oxygen: the guncotton being heated in an atmosphere of CO_2 , as in the Will test. The gas is then passed into the burette, the CO_2 and NO_2 absorbed in the potash bulb, C, and the hydrogen got rid of by washing out with oxygen and sparking as before. If the nitrogen oxides had been reduced by the guncotton the nitrogen (or nitric oxide which would give nitrogen after this process) should be detectable; but the result obtained was quite negligible even after 4 hours' heating. These experiments seem to prove that for several hours, with temperatures as low as 80°C ., a normal nitro-compound explosive does not decompose to any great extent but behaves like a dissociable substance, which gets its equilibrium with the products of dissociation. This process, however, is not alone taking place, but there is also the tendency to decompose irreversibly, which tendency is increased by rise or change of temperature, by the presence of impurities or by the production of acids. At the temperature of the Will test, where even the nitrogen peroxide is itself partially decomposed owing to the high temperature, the latter process is much more prominent. The two processes are independent; the apparent equilibrium is only an approximately constant state of affairs. Other points in the last paper have been called to notice, but these do not require so much discussion.

The reaction on the indicators.—In the above remarks, the expression "reacting gases" has been used, because it was immaterial to the argument what gas was in equilibrium with the explosive. But when interpreting the differences shown by the test paper (which is coloured by oxidation) and the drop (which detects only nitrous acid), it is important to take these differences into account. This difference was recognised, and it was considered to be an argument in favour of a "nitrous acid detecting" reagent, over the ordinary heat test paper, as with the former it becomes possible to interpret the results in a reliable manner. At the same time, Smart quite rightly points out that the starch iodide paper by reason of the back reaction ($2 \text{KOH} + \text{I}_2 = \text{KI} + \text{KOI} + \text{H}_2\text{O}$) gives a great range between good and bad, and an apparently greater sensitiveness.

Relation between concentration (C) and time (T).—In the determination of the value of K (when $\text{TC} = \text{K}$) the higher value obtained by the writer can hardly be due to depletion owing to the size of the vessel. One drop introduced into an atmosphere containing 0.017 mgrm. of nitrogen peroxide would hardly deplete it sufficiently to account for the different value of K obtained, for one drop absorbs only 4×10^{-4} mgrm. of N_2O_4 , but for times of 50' or longer, no doubt the effect is appreciable. Local depletion of NO_2 in the neighbourhood of the drop might have accounted for the difference, but it was found that stirring the gas during the absorption by the drop made little difference to the time. However the results were not satisfactory and the errors were no doubt due to condensation on the side of the vessel; this effect appeared to be more prominent than that of absorption by alkalinity of the glass, as a silica globe gave much the same results.

The other method employed was the measurement of the vapour pressure of nitrogen peroxide. The vapour pressure of nitrogen peroxide at 90° is about 0.009 m.m., the time of colouring of a drop in an atmosphere exhausted with NO_2 from nitrogen peroxide at this temperature is 11'.

The value for $\text{TC} = \text{K}$ would consequently be about 140×10^{-6} ; measurements at other temperatures below 80° , agree approximately with this value for K, which is not very different from that obtained by the other method.

Heat tests on guncotton.—A long series of experiments, which will be published in full, have all led to the idea that nitric acid, or at any rate H^+ ions, formed in the moisture in the guncotton has an accelerating influence on the decomposition, and that as the water is removed, the rate of evolution of gases becomes slower. It does not seem possible to explain otherwise the results of continuous tests, tests done after drying or evacuation, and tests done in the presence of other substances. The evolution of moisture follows a similar curve to the evolution of other gases. In looking through all the evidence for the view which has been held about the nitric acid, there does not appear to be any better hypothesis, for it fits in well with all the facts.

Continuous tests.—The suggestion made by Smart about the curious difference in the behaviour of iodide paper and of the α -naphthylamine drop at the beginning of continuous tests on cordite offers a possible explanation; but owing to change of temperature, the rate of evolution of gases is probably greater at first, when the unstable portions of the substance are broken up: the drop which absorbs more, may therefore deplete the atmosphere of gases too quickly and give rise to the anomalous maximum in the curve obtained with the drop reagent. This seems to the writer a more probable explanation, but further work would be needed to clear up this point.

Absorption of NO_2 by guncotton.—The writer pointed out that there seemed to be contradiction in the results of experiments with guncotton and nitroglycerin respectively which had been described by Robertson and Smart. The contradiction is due, in Smart's opinion, to the physical difference of the guncotton and the nitroglycerin comminuted on silica fibres. No doubt this physical difference exists, but it should tend to give an effect in the opposite direction. In the writer's opinion, the contradiction is due rather to the fact that the nitrogen peroxide is absorbed differently in the two cases; in the guncotton it is absorbed chiefly, not wholly, by the moisture in the guncotton, and in the nitroglycerin it is dissolved in that substance itself. Therein is the physical difference.

There is no doubt, however, that physical difference in the nature of explosives is a very important matter from the practical point of view. A single heat test, for this reason, often gives an unreliable result; a continuous test, on the other hand, might overcome such difficulties.

The practical use of the heat test.—In the discussion of these details, the main problem must not be lost sight of, that is, so to improve the heat test that it may become a thoroughly reliable stability test.

The vagaries of the test are due to differences in the moisture content of the explosives, the presence of foreign substances (like acetone), the physical condition of the explosive and the unreliability of the reaction on the starch iodide paper. None of these would matter, if the differences caused by each effect could be ascertained. It is the writer's opinion that the suggestion made in the last paper would attain this object, namely that a continuous test on the sample of the explosive should be carried out alongside that on a standard sample, and a comparison made of the results obtained. The continuous tests need not last more than 2 or 3 hours, and tests need only be made every $\frac{1}{2}$ hour. It is suggested, also, that the tests should be done with a nitrous acid indicator in the drop form, as well as with the iodide indicator on paper: in this way a reliable knowledge of the stability of the explosive could be established, for the effect of the various disturbing factors could be ascertained.

The values of the first test are important, because if it is short the explosive contains oxides of nitrogen from previous decomposition, or it shows its instability owing to the change of temperature to which it has been submitted; subsequent tests should make it possible to distinguish

between these two sources. A single Abel heat test is doubtless a valuable indication of the stability of the explosive, and it has the advantage of being carried out under conditions very similar to those to which the explosive is normally exposed, and it is easy to carry out; its unreliability however, owing to various influences, is its chief drawback. The writer hopes that this drawback will be removed by his suggestion.

DISCUSSION.

Dr. R. ROBERTSON understood Mr. Egerton to hold that while at some higher temperature (say at that of the Will test) the dominant action was that of decomposition, it was possible at the lower temperature of 80° C. for an equilibrium to be set up between the explosive and its products of decomposition. Apart from the known catalytic effect of these on the explosive heated under dry conditions, it might be recalled that even down to a temperature of 40° C. Vieille had found guncotton to decompose in a continuous and regular manner (*Mém. Poudres et Salpêtres*, 15). He gathered that in the apparatus illustrated on the board, less nitrogen had been obtained than might have been expected. That might possibly be due to absorption of nitric peroxide by traces of liquid water in the small space in which the products of decomposition were evolved, for he had found in experiments on the decomposition of guncotton in *vacuo* (*J. Chem. Soc.*, 91, 772) that this space must always be sufficiently large to prevent the deposition of liquid water, proceeding either from decomposition of the guncotton or constitutionally combined, or possibly oxides of phosphorus may have absorbed some of the oxides of nitrogen. In contact with heated guncotton the oxides of nitrogen were reduced to nitrogen, and the more readily the greater the opportunity for reaction between them and the heated explosive. This reduction, noticeable in the Will test, where the products were carried off in a stream of CO₂, became much more pronounced in a closed space, and might have more bearing on the results obtained in the continuous test with guncotton, than the conception of an equilibrium in an instance where the products were found to exercise a catalytic influence on decomposition and where the decomposition had been followed down to a fairly low temperature.

Mr. W. MACNAB said that up to a point the heat test was an extremely useful means of testing explosives, but when it was pushed too far and asked to do too much, he thought one got into an unknown land. On the other hand, work like that of Mr. Egerton and Dr. Robertson was all to the good, and ultimately they would get a clear idea of what the heat test could do.

Mr. EGERTON in reply said that, with regard to Dr. Robertson's remarks as to the Will test, he thought those results were not very different in a way from what he (Mr. Egerton) had got. Will in his experiments had mentioned the other irreversible effect, and as Dr. Robertson had said those experiments lasted several years. He had noticed especially with cordites that the results of his continuous tests remained constant for a time and then dropped, then remained constant a bit and dropped. He, the speaker, had never gone on for more than 36 hours even with guncotton; but if one went on long enough he had no doubt the same effect would be observed with guncotton. The apparatus he had shown was evacuated with a pump. The guncotton was admitted and the pump had a phosphorus pentoxide tube in connection with it so that the guncotton was fairly dry. After long heating the guncotton gave off the water of constitution; but he had not seen a sufficient quantity in the tube so he did not think nitrous acid could account for these results. On the other hand, he criticised the apparatus himself and he could improve it for the purpose. The mercury was bound to get left in the burette to a small extent, and of course the mercury would absorb the nitrogen oxide at once. Besides that, there was a small amount of grease in the taps which would take some nitrogen oxide, but he did not think that could account for it all disappearing. Finally, he thanked Sir William Ramsay for allowing him to use the apparatus, which was one of Sir William's masterpieces.

Manchester Section.

Meeting held at the Grand Hotel on Friday, December 5th, 1913.

MR. JULIUS HÜBNER IN THE CHAIR.

THE ACTION OF STRONG NITRIC ACID ON COTTON CELLULOSE.*

BY EDMUND KNECHT AND ADOLF LIPSCHITZ.

The remarkable action of strong nitric acid (68 per cent.) on cellulose appears to have been first noticed by Francis (Chem. Soc. Trans. 1885, 183), who proposed to utilise this action for the toughening of filter papers intended for use in filtering under diminished pressure. In 1893, Horace Koechlin pointed out that cotton on being steeped in strong nitric acid did not undergo any diminution in strength, but that it acquired an affinity for basic colours (rhodamine). He also found that after washing the fibre retained a certain amount of fixed nitrogen. In 1896, one of the present authors showed (*Soc. Dyers and Col.*, 12, 89) that when bleached cotton is treated with nitric acid of 82° Tw. it contracts considerably, becomes much stronger and evinces a greatly increased affinity for direct cotton colours; no increase in its affinity for methylene blue could, however, be observed. On the other hand it was noticed that the treated cotton showed a marked affinity for some acid colours (e.g., Brilliant Scarlet 2 R. Berlin). It was further shown in 1904 by the same author (*Ber.*, 37, 549) that the mercerising action of nitric acid on cotton only began at ordinary temperatures at 76° Tw. and that the most suitable strength of the acid for the purpose was 83° Tw. Acid of 62°, 42°, and 22° Tw. was found to exert a tendering action on the fibre. These results were partly corroborated by the researches of Hübner and Pope (this Journal, 1904, 23, 406), who showed that the amount of benzo-purpurin taken up by cotton which had been treated with acid of 65° Tw. was very small as compared with that taken up by cotton which had been treated with acid of 83° Tw.

It was further shown by one of the present authors (i.e.) that the mercerising action was preceded or accompanied by the formation of a labile cellulose nitrate which approximated to the composition C₆H₁₀O₂.HNO₃ and which was instantly decomposed by contact with water into nitric acid and cellulose, showing in this respect an analogous behaviour to the phenanthrenequinone nitrate of Kehrman and Mattisson (*Ber.*, 1902, 35, 343).

It has been shown by Vieille (*Compt. rend.*, 1882, 95, 132) and by Häussermann (*Z. ges. Schiess- u. Sprengst.-Wesen*, 1908, 3, 121) that by the action of nitric acid of sp. gr. 1.52 on cellulose, actual nitration takes place, with the formation of a nitrate containing as much as 12.65 per cent. of nitrogen, i.e., an amount approximating to that of a fully nitrated product (guncotton).

According to C. Hoitsema (*Z. angew. Chem.*, 1898, 173) it is not possible to obtain a more highly nitrated product than that corresponding to the formula C₁₂H₁₄O₂(NO₃)₂, which demands 14.14 per cent. of nitrogen.

According to H. Koechlin, cotton treated with ordinary concentrated nitric acid forms a cellulose dinitrate, and this was corroborated by Knecht (i.e.). It has however, since been ascertained that the amount of fixed nitric acid in the treated cotton is considerably less than that demanded by a dinitrocellulose. The former conclusions of one of the authors were based mainly on the results of nitrogen estimations by the Dumas method, and it is probable that the copper spiral employed was not long enough, for subsequent experiments showed that it is necessary, in the estimation of nitrogen in nitrocelluloses,

* Excerpt from the dissertation of A. Lipschitz, presented for the degree of Doctor of Technical Sciences at the Federal Technical High School, Zürich.