of sulfur present) are sufficient. To give an idea of the results to be had by the other three methods, Table IV has been included. These data show the results which were obtained under conditions distinctly unfavorable for accurate results, only 6 to 30 mg. of sulfur and large and varying amounts of ammonium carbonate being present in the samples used. These conditions were chosen as representing those with which we had to deal in the gas testing; and if in other work larger amounts of sulfur were present or greater care was exercised no doubt much more exact results could be had. For gas testing the accuracy here shown is ample, since it is greater than the accuracy obtainable in the condensation of the sulfate.

TABLE IV—COMPARISON OF METHODS FOR SULFATE DETERMINATION. SULFUR (MG. PER G. OF SOLUTION) IN STANDARD H_2SO_4 SOLUTION DETERMINED BY DIFFERENT METHODS IN PRESENCE OF EXCESS OF AMMONIUM CARBONATE EXCEPT AS OTHERWISE NOTED

Volumetric against NaOH and benzoic acid	Method 1 Gravimetric	Method 2 Gravimetric	Method 3. Volumetric	
			Oxalate standard (no carbonate added)	H_2SO_4 standard
1.589	1.591	1.597	1.602	1.595
93	91	93	1.597	1.602
90	90	92	1.600	1.597
92	1.609(a)	99	1.599	1.601
92	1.596	97	1.602	1.596
92	96	92	...	1.589
89	88	99	...	1.574
90	92	91	...	1.575
..	94	1.600	...	...
Av.	1.591	1.592	1.595	1.600
				1.589

(a) Omitted from average.

The first set of results in Table IV by Method 3 are calculated on the basis of the value of the thiosulfate solution obtained by titration against a permanganate solution, whose value was obtained against sodium oxalate. In the second series the thiosulfate was standardized against the sulfuric acid value given in the first column of the table.

For accurate work the gravimetric methods, with the precautions which have been carefully developed by many experimenters, are to be preferred; but for rapid work the turbidimetric and volumetric methods are very useful. They can be applied in many lines of work other than gas testing, since in each the value of the standard is fixed against similar material whose value has been previously established by other methods.¹ If the calibration and testing procedures are identical the variation of individual tests from the average or correct value is the important factor; this variation has been shown by our experience to be about one per cent or less in the volumetric and five per cent or less in the turbidimetric method.

BUREAU OF STANDARDS
WASHINGTON

THE DETERMINATION OF SULFUR IN ILLUMINATING GAS¹

By R. S. MCBRIDE AND E. R. WEAVER

The methods for determination of sulfur in gaseous

¹ Paper presented at the Annual Meeting of the American Chemical Society, Milwaukee, March, 1913. Published by permission of the Director of the Bureau of Standards. This paper is a report on tests made at this Bureau, preparatory to the recommendation of a method for official inspection of gas; a more complete report on this same investigation is to appear soon in Technologic Paper No. 20 of the Bureau.

fuels can be classified in four groups, the general principles of which are as follows:

(1) The gas is burned and the sulfur oxides in the products of combustion are condensed or absorbed, oxidized to sulfate, and determined as such.

(2) The sulfur compounds are oxidized by liquid reagents giving the sulfate, which can be determined directly.

(3) The sulfur compounds are oxidized by burning the gas but the sulfur is absorbed and determined as sulfur dioxide.

(4) The sulfur compounds are reduced to give hydrogen sulfide, which is then absorbed and determined.

The methods of Group 1 have been most used in American practice and are generally considered the most accurate and convenient, either for works control or official testing. Tests have been made on five forms of apparatus of this group and one or more forms of each of the other classes; but the principal investigation was limited to the three forms, all of group one, which appeared to be most satisfactory for general use. These are known, respectively as the Referees apparatus, the Hinman-Jenkins apparatus, and the Elliott apparatus.

The Referees apparatus is too well known to require a detailed description here. It consists of a Bunsen burner surrounded by pieces of crystallized ammonium carbonate, from which the products of combustion of the gas, mingled with vaporized ammonium carbonate, pass into a condensing cylinder where the sulfur oxides are absorbed in the water condensed from the products of combustion and are oxidized, in the presence of the ammonia, to ammonium sulfate.

The Elliott apparatus is essentially a large Referees apparatus, the principal difference being the use of two condensing cylinders through which the gases successively pass. The Hinman-Jenkins apparatus² differs from the Referees greatly in form, but in principle only in the use of concentrated ammonium hydroxide instead of ammonium carbonate as a source of ammonia.

To compare the several forms of apparatus, two or more were operated at the same time, being supplied with gas from a common source. Suitable precautions to maintain a uniform supply to each apparatus and to prevent errors due to sulfur in the air of the room were taken. The meters used were frequently compared to determine their relative accuracy.

One series of comparisons made upon the three apparatus to determine the best conditions of operation and sources of error is summarized in Table I. The results tabulated include only those obtained under normal working conditions, and not those obtained when variations were made which would be avoided in practice. The sulfate in the condensed liquids was determined as barium sulfate by a method closely resembling that of Johnston and Adams,² the weight

¹ *J. Am. Chem. Soc.*, **28**, 543 (1906).

² *Ibid.*, **33**, 829-45 (1911).

of the precipitate being corrected for silica dissolved from the glass by the alkaline liquids.

The cause of the lower results obtained by the use

TABLE I—FINAL COMPARISON OF REFEREES, ELLIOTT AND HINMAN-JENKINS APPARATUS

A. Gas burned at the rate of: Referees 0.60, Elliott 0.96, Hinman-Jenkins 0.60 cu. ft. per hour.

Test No.	Refer-ees No. 1: Sulfur per 100 cu. ft.	Elliott: Sulfur per 100 cu. ft.	Ratio Elliott: Refer-ees No. 1	Hin.-Jenk.: Sulfur per 100 cu. ft.	Ratio Hin.-Jenk.: Refer-ees No. 1	Ratio Elliott: Hin.-Jenk.
10	11.3	10.9	98.2	..	...	...
11	13.8	14.3	103.6	13.8	100.0	103.7
12	13.9	14.8	106.6	14.7	106.0	100.8
13	13.4	13.9	104.1	13.7	103.3	101.3
14	13.4	12.6	93.8	14.0	104.7	90.0
15	13.5	14.8	109.9	14.0	103.7	106.0
16	13.8	...	...	14.0	101.0	...
17	13.6	...	...	13.8	101.5	...
Av.			102.7		102.9	100.3

B. Gas burned at the rate of: Referees 0.49, Elliott 1.20, Hinman-Jenkins 0.65 cu. ft. per hour.

18	15.2	14.9	98.0	14.3	94.0	104.1
19	13.9	14.0	101.0	13.0	93.6	107.8
20	14.3	...	...	13.3	93.0	...
21	15.4	...	...	14.4	93.5	...
22	13.1	13.1	100.0	12.9	98.5	101.7
23	13.2	...	...	12.2	92.5	...
Av.			99.7		94.2	104.4

C. Gas burned at the rate of: Referees 0.50, Elliott 1.20, Hinman-Jenkins 0.50 cu. ft. per hour.

24	13.8	15.0	108.5	13.9	100.7	107.9
25	10.3	9.9	96.0	9.9	96.0	100.0
26	16.1	16.6	103.0	...	...	...
27	13.5	...	...	13.9	102.6	...
28	11.6	...	...	12.1	103.9	...
29	13.2	...	...	13.0	98.3	...
Av.			102.5		100.3	103.9

of the Referees apparatus was next investigated. It was found that by the addition to the Referees apparatus of a second condenser, which practically doubled its condensing surface, the amount of sulfur found was increased by about 1 per cent.

The effect of varying the rate at which the gas was burned, shown in Table I, is still better indicated in Table II, which shows the percentage of sulfur condensed in each tower of the Elliott apparatus with the gas burning at three different rates. The significance of the results appears at once when we remember that the first tower with its connections is practically a large Referees apparatus and the sulfur which passes into the second tower is lost in the Referees form.

From our experiments we believe that for each apparatus there is a certain rate of gas consumption above which the loss of sulfur increases out of all proportion to the increased rate at which the gas is burned. If the ammonium carbonate or hydroxide is renewed as frequently as is recommended, one cubic foot per hour for the Elliott and one-half cubic foot per hour for the other forms of apparatus are safe rates; but the loss increases rapidly at higher rates. Indeed, the amount of sulfur condensed per hour in the Elliott apparatus was less when the gas was burning 1.36 cu. ft. per hour than when burning at 1.20 cu. ft. per hour.

Our work throughout indicated that the most

TABLE II—AMOUNT OF SULFUR CONDENSED IN DIFFERENT PARTS OF THE ELLIOTT APPARATUS AT DIFFERENT RATES OF GAS CONSUMPTION

Rate (cu. ft. per hr.)	Sulfur in 1st tower (grains per 100 cu. ft. of gas)	Sulfur in 2nd tower (grains per 100 cu. ft. of gas)	Total sulfur (grains per 100 cu. ft. of gas)	Per cent of total condensed in sec-ond tower	Referees burned at 0.5 cu. ft. per hr. (sulfur, grains 100 cu. ft. of gas)
1.36	5.7	3.7	9.4	39	15.5
..	8.2	4.7	12.9	36	14.2
..	4.9	1.5	6.4	23	11.5
1.20	13.9	1.1	15.0	8	13.8
..	9.4	0.5	9.9	6	10.3
..	14.8	1.8	16.6	8	16.1
0.9	11.5	1.7	13.2	1	13.7
..	10.4	3.9	14.3	3	12.8
..	10.7	2.5	13.2	2	12.4
..	12.1	1.6	13.7	1	13.6
..	12.2	2.0	14.2	1	13.8
..	13.1	2.1	15.2	1	15.0

serious source of error in any apparatus of this type is an insufficient or irregular ammonia supply.

Table III shows the effect of using in the Referees apparatus lumps of carbonate which had previously been used and which were, therefore, incrustated with bicarbonate. As much ammonium carbonate was used in each case as would lie in the annular space about the burner. From the decrease in efficiency of condensation on long use of the carbonate, shown by the results in Table III, it appears that a 10-hour run, using a single supply of carbonate, could hardly be expected to give more than 90-95 per cent or a 20-hour run more than 75-80 per cent of the sulfur burned.

TABLE III—EFFECT OF USING OLD AMMONIUM CARBONATE ON REFEREES APPARATUS

Test No.	Previous use of carbonate (hours)	Length of run (hours)	Sulfur condensed with old carbonate (grains) A	Sulfur condensed with fresh carbonate (grains) B	Ratio of A to B
30	6.0	6.5	7.0	14.0	0.50
31	6.0	6.5	11.5	14.9	0.77
32	6.5	6.0	9.5	13.9	0.78
33	6.0	6.0	11.0	13.6	0.81
34	No carbonate used		5.1	16.8	0.30

Table IV shows the effect of varying the ammonia supply to the Elliott apparatus upon the percentage of the total sulfur which escapes from the first tower and is collected in the second. Similarly, Table V shows the percentage of total sulfur collected in the added condenser of the Referees apparatus previously mentioned under Table I: *first*, when nothing was added to this second condenser; *second*, when concentrated ammonium hydroxide was slowly dropped in at the top of the second condenser; *third*, when a solution of hydrogen peroxide and ammonium hydroxide was added in the same way. The object of adding hydrogen peroxide was to see what effect an oxidizing substance would have upon the absorption of the sulfur dioxide; from the results obtained this effect appears to be negligible.

Another series of experiments showed that alkalinity of the atmosphere as distinguished from alkalinity of the condenser surface was necessary for proper condensation of the sulfur.

SUMMARY

A comparison of the Referees, Hinman-Jenkins, and

Elliott apparatus for the determination of sulfur in gas showed that any of these apparatus is capable of giving satisfactory results if properly operated.

TABLE IV—PERCENTAGE OF TOTAL SULFUR CONDENSED IN SECOND TOWER OF ELLIOTT APPARATUS BURNING 1.2 CU. FT. OF GAS PER HOUR

(NH ₄) ₂ CO ₃ renewed every three hours	(NH ₄) ₂ CO ₃ renewed every hour	Air supplied to apparatus bubbled through conc. NH ₄ OH
7.5	0.6	0.1
6.0	0.8	...
8.0	...	...

TABLE V—PERCENTAGE OF TOTAL SULFUR CONDENSED IN ADDED CONDENSER OF REFERREES APPARATUS

Nothing added to second condenser	NH ₄ OH added to second condenser	NH ₄ OH + H ₂ O ₂ added to second condenser
1.0	3.6	1.2
1.1	2.2	2.3
0.8	0.9	2.0
...	...	1.0
Average 1.0	2.2	1.6

Three important conditions for successful operation are: (1) A proper rate of combustion; (2) a strongly alkaline atmosphere in the condenser chamber; and, (3) elimination of all rubber connections between the burner and condenser.

BUREAU OF STANDARDS
WASHINGTON

SOME RESULTS OF THE HYPOCHLORITE DISINFECTION OF THE BALTIMORE CITY WATER SUPPLY¹

By J. BOSLEY THOMAS AND EDGAR A. SANDMAN

Stokes and Hachtel² have reported the results obtained by the hypochlorite disinfection of the Baltimore City water supply during a period extending from the institution of the treatment on June 15, 1911, to October 30, 1911. They examined samples taken from the untreated water in the impounding reservoir and from the treated water after it had passed through each of two storage reservoirs. The results of their examinations showed bacterial reductions varying between 94.5 and 99 per cent. They also showed average reductions in the *Colon bacillus* from 57.5 per cent positive tests with 0.1 cc. of untreated water to 12 per cent positive tests with 0.1 cc. of treated water, and from 89 to 40 per cent with 1.0 cc. The greatest reductions were obtained with one part per million of available chlorin, when there were shown reductions from 86 per cent positive tests with 0.1 cc. of untreated water to 9.5 per cent positive tests with 0.1 cc. of treated water, and from 100 per cent to 37 per cent with 1.0 cc.

The period covered by the following report extends from January to December, 1912. In addition to the places sampled by Stokes and Hachtel, we obtained samples at the influent of the first storage reservoir, after the water had passed through seven miles of tunnel subsequent to treatment. The time required for the water to pass through this tunnel varies between 4.9 and 12.2 hours. While allowing sufficient time for effective disinfection, the taking

of samples just before the water enters the first storage reservoir permits of counts being obtained before any after-growths are likely to have occurred.

The amount of available chlorin applied during the period covered by the report of Stokes and Hachtel was raised from 0.4 part per million, applied at the start on June 15, to 0.6 on June 23, and to 1.0 on October 15. On July 15, 1912, the amount was again raised, by order of the Commissioner of Health, to 1.5 parts per million, and this amount has been maintained until the present time.

From June 11 to November 12, 1912, aluminum sulfate, in amounts varying between 0.61 and 1.05 grains per gallon, was applied to the water as it entered the first storage reservoir.

Shortly after the period covered by the report of Stokes and Hachtel, after-growths in the storage reservoir caused excessive bacterial counts. These conditions maintained during the first five months of the year, but about the middle of May the counts showed a marked diminution, and no further after-growths were observed, excepting during a few days in September.

The monthly averages of the results in bacterial counts and *B. coli* tests, shown in an accompanying table, are taken from daily analyses. The counts during the first six months were obtained on standard agar at 20° C., and during the remainder of the year at 37° C. The *B. coli* averages were obtained from tests made on portions of water varying by a multiple of ten from 0.001 cc. to 100 cc., sufficient number of tubes being used in each case to secure at least one negative and one positive test, excepting when no fermentation was obtained with 100 cc. The average number of *B. coli* per cc. for each month was estimated by considering the number of positive and negative tests in each dilution and following the method described by Phelps before the American Public Health Association in 1907. Lactose bile was used as an initial medium, and Endo's agar was used for isolating the members of the *B. coli* group in pure culture, nearly 100 per cent successful isolations having been obtained by the use of this medium, whereas the frequent encountering of spreaders on litmus agar and the fact that many of the acid-forming colonies proved not to be members of the *Colon* group seriously impaired the efficiency of this latter medium. No attempt was made until in the last two or three months to differentiate the four members of the *Colon* group; but this is now being done with the use of dulcitol, in addition to the usual sugars, and morphological examinations, and the results seem to show a greater vulnerability of the two *B. coli* organisms than of *B. aerogenes* and *B. acidi lactici*.

The results obtained by the use of the 20° temperature show much greater reductions in the bacterial count than those obtained with the 37° temperature, and we believe that counts should be made at the higher temperature in addition to those made at 20°.

The effects of the treatment of this water supply have been a very good reduction in the bacterial count of the water as it enters the first storage reservoir,

¹ Paper presented at the Annual Meeting of the American Chemical Society, Milwaukee, March, 1913.

² *Am. J. Pub. Health*, April, 1912.