# DETERMINATION OF TOTAL SULPHUR IN ILLUMINATING GAS.

BY CHARLES D. JENKINS.

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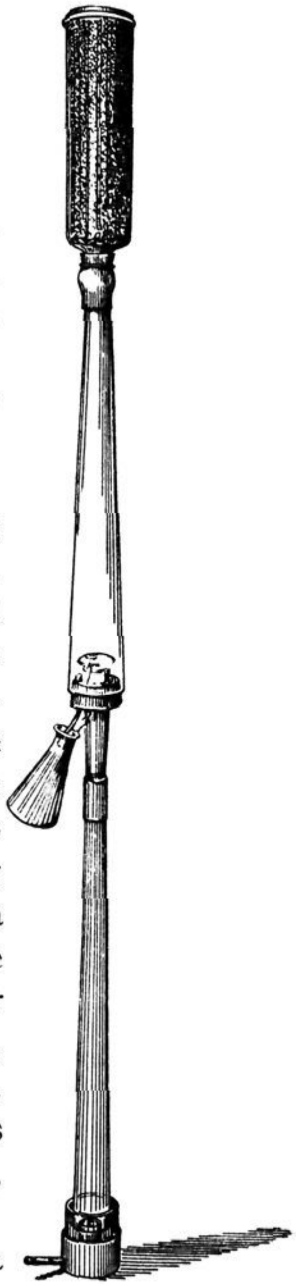
THE apparatus here described has been in use in Massachusetts for some years for obtaining the amount of sulphur, in all forms, in illuminating gas. A feature of the apparatus, which in its original form was devised by C. W. Hinman and since improved by the writer, is portability; in its present form only three or four minutes are required to set up or take down the apparatus ready for traveling.

The upper vessel is a "bead glass" 300 mm. long and 60 mm. in diameter; this is filled with large cut glass beads, held up by a suitable fluted glass, giving a large condensing surface without obstructing the draft. To this bead glass is attached, by a rubber connector, the upper adapter, 410 mm. long and 50 mm. lower, internal diameter. To the upper adapter is attached, by means of the "connecting piece,', the lower adapter, 400 mm. long and 40 mm. lower diameter. The connecting piece projects 12 mm. above the top of a rubber stopper, fitting the upper adapter, and is surmounted by a watch-glass deflector, carried on platinum wires. An overflow tube carries the condensation to the Erlenmeyer flask hung on the stopper as shown; this tube is so adjusted that some liquid remains on the stopper, to keep it cool and to absorb some of the ascending gases. The Bunsen burner is fitted with a lava tip having a 5 mm. hole; surrounding the burner is a glass tube 20 mm. in diameter forming the inner wall of an annular chamber, of which the outer wall is a glass ring 50 mm. in diameter. Into this chamber, which serves to contain 10 per cent. ammonium hydroxide, the lower adapter dips 10 mm.



The gas to be tested is measured through a small dry gas meter and then burned at the bottom of the system of condensers at the rate of 0.6 cubic feet per hour. All the air supplied to the burner mixing tube and to the flame must pass over the ammonia in the annular chamber, thus insuring an ammoniacal atmosphere throughout the apparatus. Ten per cent. ammonium hydroxide is added at the rate of 2–3 cc. for each 0.2 cubic foot of gas burned. The various sulphur compounds burn to sulphur dioxide and sulphur trioxide, which, combined with the ammonia, are dissolved in the condensed water and collected; the apparatus is washed out with 200 cc. of distilled water.

The sulphur is determined as sulphuric acid by a volumetric chromic acid method worked out by Hinman[1] in 1877. Andrews[2] and Pennock[3] have described a similar method. To the solution and washings obtained from burning 0.8 to 1.0 cubic foot of gas are added 2-3 cc. bromine water and the solution evaporated to 30–40 cc. in bulk. To this hot solution is added an excess of a hydrochloric acid solution of barium chromate; it is gently boiled, an excess of dilute ammonia added, boiled again for a minute, filtered and washed. The ammonium chromate in the filtrate (the chromic acid being equivalent to the sulphuric acid in the original solution) after being boiled in a stout flask with a Bunsen valve, to expel air, is cooled and titrated direct with stannous chloride, using the iodo-starch blue to accentuate the end-point.

A weak sulphuric acid was analyzed by different methods with the following results:

| | Gram. |
|---|---|
| The usual gravimetric method gave | 0.04772 |
| Titrating against sodium carbonate | 0.04783 |
| The above volumetric method gave | 0.04804 |

Four sets of apparatus were run in parallel with a common gas supply. The sulphur being determined as above, gave grains per 100 cubic foot of gas: 12.26, 12.29, 12.23, 12.26.

Ten cubic feet of gas were burned in several apparatus and the sulphur determined, in duplicate, gravimetrically and volumetrically, as follows:

| | Grains per 100 cu. ft. | Grains per 100 cu. ft. |
|---|---|---|
| Gravimetrically | 10.97 | 10.97 |
| Volumetrically | 11.01 | 11.04 |

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[1] Amer. J. Science and Arts, 14, 478.

[2] Am. Ch. J. 11, 567; 32, 476.

[3] This Journal, 25, 1265.

With this apparatus and method of analysis sulphur in illuminating gas can be rapidly and accurately determined in a small sample of gas.

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# A SYSTEM OF RECORDS FOR ANALYTICAL LABORATORIES.

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A SATISFACTORY system of records for an analytical laboratory requires that all the data concerning each sample which is analyzed shall be in one place, that this information may be easily found when needed, and that the records shall be in some compact form which can be conveniently preserved in some indestructible and fire-proof place. A further convenience is attained if the record of the sample and the results of its analysis appear together in some complete and concise form. A great saving of time and labor is insured if the records are so arranged that the terms used in recording and reporting results of analyses are in printed form, thus requiring only the filling in of the analytical numbers in their proper places. A system which embodies all these advantages was devised by the writer and has been in use in the chemical laboratory of the Washington Agricultural Experiment Station for the past two years, and has given such excellent satisfaction that a brief description of it is offered below with the idea that its use may be of very general application and advantage.

The general plan of these records is a modification of the card index system which is so universally used. A set of cards, five by eight inches in size, was obtained. On one face of each card was printed a blank form for recording the origin of the sample, its description, the date of its receipt, the date of analysis, and the signature of the analyst; and beneath this the statement of the results of the analysis. The set of cards in use in this laboratory is printed in five different forms as shown in the accompanying illustration. These forms are appropriately printed for recording analyses of "Soils," "Fertilizers," "Feeding Stuffs," "Foods" and "Miscellaneous" samples. Each form is printed on cards of a different color, a great convenience in