ART. XVII.—*Description of a new Apparatus for Gas Analysis*;
by C. W. HINMAN.

SOME time since, having occasion to make some analyses of illuminating gas, I read descriptions of several forms of apparatus for that purpose, but failed to find one which appeared quite satisfactory.

An apparatus was desired which should be as far as possible free from fragile or costly parts, and which, without being too complicated, should require no corrections to be made for variations in the pressure, temperature, or aqueous vapor; reliable results rather than minute accuracy being desired.

The apparatus finally adopted operates on the same principles as Williamson & Russell's,* except that in my apparatus the gas is not exploded in the measuring tube, but in a bulb for that special purpose. The trough is nearly the same as that of Doyère,† and pipettes are also used.

The apparatus as made consists of a measuring tube *a*, about 230 mm. long, about 20 mm. in diameter, divided into fortieths of an inch and calibrated with mercury as described by Bunsen. The tube is firmly held by a clamp on the end of the rod *b*, which rod slides up and down in *c*, and is clamped in any position by the screw *d*. A slow motion is given to *c*, and thus to the measuring tube, by means of the milled headed nut *e*, which works along a thread cut on the rod *f*, which is firmly secured

* Chem. Soc. J., [2] ii, 238.

† For figures and description of Doyère's Eudiometer, see Ad. Wurtz Dictionnaire de Chemie, I, p. 280, fig. 42.

to the body of the apparatus. The screw *g* can be turned in so that its end just fits into a longitudinal slot in the rod *f*. By this means *c* is prevented from turning around *f*, and the measuring tube can thus be kept exactly over its well: or *g* can be used to clamp *c* in any position. The pressure tube *h* is about 200 mm. long, and has the same diameter in the upper part as the measuring tube: the lower part, which is about 50 mm. long, has a diameter of only 6 mm. A mass of lead, covered with sealing wax, is attached to the lower part of *h*, so that when it is filled with air, it will keep upright when it is suspended from an eye at the top. The pressure tube is hung by a wire from the end of the arm *i*, which slides with friction along the rod *f*.^{*} The mercurial trough is of cast iron, and consists of the plate *l*, which is about 130 mm. square, 15 mm. thick, and has a groove in its upper surface 10 mm. wide, 6 mm. deep, and 5 mm. from the edge; the well *k*, which is about 240 mm. deep and 30 mm. in inside diameter; and the side well *m*, for the pipette, which is of the same depth as *k*; but part of it extends 40 mm. above the top of *l*; *m* is 10 mm. wide and 90 mm. broad. The wells *m* and *k* have walls 6 mm. thick. Of course *k*, *l* and *m* are all cast in one piece. The groove in *l* has cemented into it, by means of a cement composed of beeswax and rosin, a rectangular trough of plate glass 5 mm. thick, cemented with the same cement into a sheet-iron frame *j*, which is made by bending a sheet of the right size into a box without ends, and then cutting out the sides so as to leave only a strip 12 mm. wide on each edge. The legs *n* are screwed on to the plate *l*.

The pipette consists of a bulb *o*, of a little larger capacity than the measuring tube, open at one end and at the other melted to a tube about 5 mm. in diameter, having a bore of 1 mm., bent so that when it is put into the side well *m*, the point of the pipette can be brought directly under the measuring tube. The other end of the bulb is joined to the end of a piece of good rubber tubing, about 300 mm. long, with rather thick walls and small bore, the other end of which is connected with a small syphon which dips to the bottom of a strong glass bottle *p*, of rather larger capacity than the bulb *o*. Immediately below the bulb is a screw clip *q*. The pipette is held in a stand devised by my assistant, Mr. Lewis. It consists of two discs of wood *rr* (cut away so as to fit the bulb), to which are screwed three rods of iron *ss*, so as to hold the bulb between the discs and at the same time form a tripod to support the pipette. The pipette tube is supported by being attached to a semi-cylindrical piece of metal *t*, which is fastened into the upper disc *r*. The feet of the tripod are screwed into a disc *r'*. This style of

^{*} Compare Dr. Wolcott Gibbs, On a simple method of avoiding observations of temperature and pressure in Gas Analysis, this Journal, II, *xliv*, 376, 1869.—Eds.

pipette is to hold reagents for absorption. I have invented another sort of pipette in which to explode gases. The bulb is of the same capacity as the former one, but the sides are 12 to 15 mm. thick, and instead of the pipette tube being melted on to the bulb, it is ground into the top with emery and then cemented in with shellac containing a little Venice turpentine. Two grooves are made in opposite sides of the end of the pipette tube where it is ground into the bulb, in which are platinum wires, the ends of which approach within 1 mm. of each other. Care is taken to have the form of the pipette such that all the gas will be expelled from the bulb when the mercury rises.

The apparatus is used in the following manner: Clean mercury is poured into the well until it is about 8 mm. deep above the top of *l*. A drop of water is put in the end of the pressure tube, which is suspended, so that the bottom nearly touches *l*, and water of the temperature of the room is poured in until the glass vessel is nearly full. Air is then taken out of or added to the pressure tube until the mercury in the lower part of the tube is just on a level with the top of a straight-edged piece of glass *u*, which is fixed just behind the pressure tube and measuring tube, so that its top edge is parallel to and about 15 mm. above the top of *l*. The measuring tube being thoroughly cleansed, and a drop of water spread over the sides, is fixed in the clamp; a thin slice of cork being placed between each side of the clamp and the tube. The tube is then placed upright in a small, long-handled cup of mercury, like that used by Doyère, and is lowered through the water into the mercury well. The piece *c* is then put over *b*, and fixed in position on the rod *f*. A clean pipette, full of mercury, is then lowered into the trough and the point brought directly under the measuring tube, which is then lowered so that the pipette touches the top of the measuring tube. The bottle *p* is placed on the table, the screw clip *q* loosened and the air is forced over into the bulb *o* of the pipette. When the pipette tube has become filled with mercury, the measuring tube is raised and the pipette withdrawn. The air in the pipette is then displaced by mercury, and if the gas to be analyzed can be taken from a rubber tube, the end of this is slipped over the pipette tube and the gas then drawn in. The pipette is put into the well, placed under the measuring tube, which is lowered a little, and the gas forced into it by raising the bottle *p* to the position *p'* and opening the clip *q*. If desirable, the gas can be bubbled directly into the measuring tube by means of a properly bent tube. When as much gas as is desired is in the measuring tube, the pipette or other tube is withdrawn, the measuring tube raised or lowered until the top of the mercury in it is on a level with the top of the glass *u*, and if the mercury in *h* is not at the

same height it is adjusted by altering the height of the mercury in the trough, or by adding hot or cold water to that surrounding the tubes; care being taken to thoroughly mix the water by agitation with a stirrer. The gases in the measuring and pressure tubes are thus kept at the same pressure and temperature and are saturated with moisture. The reading of the measuring tube is effected by means of a telescope fixed at some distance from the apparatus. As the mercury is always at the same height, the telescope is always in the same position. When it is desired to subject the gas to the action of any reagent, a pipette containing a few drops of the reagent, the rest of the pipette being filled with mercury, is introduced into the trough and the gas drawn over as before described, and is then shaken up with the reagent. When the gas is to be transferred back to the measuring tube, mercury is drawn through the tube to remove any traces of the reagent, as it is essential that none of the reagent should be transferred with the gas. The point of the pipette being under the measuring tube, the bottle *p* is raised, the clip *q* opened, and the gas is forced into the measuring tube. When the gas is nearly over, the clip *q* is nearly closed, so that the gas passes quite slowly; the pipette is raised by a block of wood under its base, so that its point is 40 or 50 mm. above the surface of the mercury in the measuring tube. The progress of the liquid in the pipette tube must be watched carefully, and when it is about 20 mm. from the end, the clip *q* is closed, and by carefully pinching the rubber tube above the clip the liquid is forced within 1 or 2 mm. of the point of the pipette tube; the point of the pipette is then brought under the surface of the mercury, by raising the measuring tube, and the pipette withdrawn. The quantity of gas left in the pipette tube would be quite unmeasurable if it was five times larger.

It is convenient to have a pipette for every reagent. The pipette for explosions is manipulated in the same manner; the gases being exploded by an electrophorus. This explosion pipette, in many cases, enables the short measuring tube to take the place of a much longer one, and still be as accurate; since explosions can be made with just the requisite quantities of oxygen and the combustible gas, without any dilution. I have exploded illuminating gas and oxygen, and hydrogen and oxygen without any dilution, and I presume the bulb would resist much greater pressure, without fracture. I have found it easier to read the measuring tube, by having a piece of white paper pasted on the glass back of it and about 3 mm. above the top of the glass *u*. The pipettes used permit the complete transfer of the gases each way without the possibility of leakage, and they keep the reagents completely out of the measuring tube, where besides dirtying the surface of the mercury and thus rendering the reading uncertain, they might subsequently act injuriously

on the gas. Thus if any caustic potash was left in the measuring tube and any carbon compound exploded with oxygen, there would be found too little carbon dioxide, as part of it would be absorbed immediately after it was formed and before it could be measured. The only part of the apparatus that can be called delicate is the pipette tube, as it is rather long and comparatively unsupported. But with ordinary caution there is little danger of its breaking. If the apparatus had been constructed one-half longer than it is, its accuracy would have been increased and the system would have been nearly as manageable. This kind of apparatus is especially suited to short measuring tubes, for with long ones the pipette becomes fragile and unmanageable, besides being liable to have air drawn in through the rubber tube. The metal work is all of iron. Most of the work was done by myself and assistant. The only work that is difficult is in joining the tube to the bulb of the explosion pipette. The apparatus needs about twenty kilogrammes of mercury to work to advantage with four absorption pipettes.

The following analyses were made with this apparatus. They show the observations required and the manner of making the calculations.

Illuminating gas manufactured by the Boston Gas Light Co., and of an illuminating power of about eighteen candles. The two analyses here given were made with the same sample.

1ST ANALYSIS.		2ND ANALYSIS.	
Divisions.	Volumes in C.C.	Divisions.	Volumes in C.C.
147·3	22·90	149·7	23·26 Gas taken.
146·7	22·82	149·2	23·19 Carb. dioxide, absorbed by KOH.
146·5	22·79	149·0	23·16 Oxygen, absorbed by pyrogallate.
138·2	21·58	140·3	21·88 Hydrocarbons, " SO_3 in H_2SO_4 .
326·4	48·91	328·0	49·14 Oxygen added.
84·9	13·72	82·8	13·41 Exploded.
8·8	2·49	5·9	2·06 Carbon dioxide absorbed.
64·0	10·65	45·3	7·88 Hydrogen added.
23·0	4·59	14·2	3·28 Exploded.

1st.		Volumes. Per cent.	
·47 N_2	(A) $\text{CH}_4 + \text{CO} + \text{H}_2$	=21·11	22·90 100·00 Gas taken.
6·06 H_2O formed	(B) $\text{CH}_4 + \text{CO}$	=11·23	·08 ·35 Carbon dioxide.
2·02 O_2 left	(C) $2\text{CH}_4 + \frac{1}{2}\text{CO} + \frac{1}{2}\text{H}_2$	=25·31	·03 ·13 Oxygen.
27·33 " added	$2\text{C}-\text{A}=3\text{CH}_4$	=29·51	1·21 5·29 Hydrocarbons.
25·31 " used	$\text{A}-\text{B}=\text{H}_2$	= 9·88	9·84 42·97 Methane (CH_4).
11·23 CO_2 formed	$\text{B}-\text{CH}_4=\text{CO}$	= 1·35	1·39 6·06 Carb. monoxide.
21·11 Combustible gas			9·88 43·15 Hydrogen.
			·47 2·05 Nitrogen.

2nd.		Volumes. Per cent.	
·53 N_2	(A) $\text{CH}_4 + \text{CO} + \text{H}_2$	=21·35	23·26 100·00 Gas taken.
4·60 H_2O formed	(B) $\text{CH}_4 + \text{CO}$	=11·35	·07 ·30 Carbon dioxide.
1·53 O_2 left	(C) $2\text{CH}_4 + \frac{1}{2}\text{CO} + \frac{1}{2}\text{H}_2$	=25·73	·03 ·13 Oxygen.
27·26 " added	$2\text{C}-\text{A}=3\text{CH}_4$	=30·11	1·28 5·50 Hydrocarbons.
25·73 " used	$\text{A}-\text{B}=\text{H}_2$	=10·00	10·04 43·16 Methane (CH_4).
11·35 CO_2 formed	$\text{B}-\text{CH}_4=\text{CO}$	= 1·31	1·31 5·63 Carb. monoxide.
21·35 Combustible gas			10·00 42·99 Hydrogen.
			·53 2·28 Nitrogen.

The hydrocarbons gave 17.70 per cent carbon dioxide by a subsequent determination. The calculations show that an error in the nitrogen determination affects the carbon monoxide, methane and hydrogen by the amount of the error. If in the place of the nitrogen found in the first analysis, we substitute that found in the second, we have CH_4 43.20 pr. ct.; for CO 5.85 per ct.; and for H_2 42.93 per ct.; and these numbers agree much better with the numbers of the second analysis. I have not been able to find any duplicate analyses of coal gas and not many of any kind, so I am not able to compare the accuracy of these analyses with that of analyses made with a different apparatus. Each of these analyses required from three to four hours for its execution.

Analyses of common Air.

Air collected April 25, 1874, freed from CO_2 .

210.2	32.07	Air,	H_2O formed	20.15
317.2	47.57	H_2 added,	O_2	6.716
178.2	27.42	Exploded,	per cent of O_2	20.94

Same Sample.

197.7	30.25	Air,	H_2O formed	19.00
304.7	45.75	H_2 added,	O_2	6.333
173.6	26.75	Exploded,	per cent of O_2	20.93

Air collected April 26.

214.3	32.66	Air,	H_2O formed	20.52
311.9	46.81	H_2 added,	O_2	6.84
170.5	26.29	Exploded,	per cent of O_2	20.94

Same Sample.

207.7	31.70	Air,	H_2O formed	19.90
306.1	45.95	H_2 added,	O_2	6.633
168.8	26.05	Exploded,	per cent of O_2	20.92

Each of these air analyses required about half an hour for its execution. These analyses seem to have about the same degree of accuracy as the average of those made by Bunsen.

Gas generated by action of KOH on Fe and Zn.

	Div.	C.C.		
Air	193.5	29.64	18.47 H_2O formed	
Gas added	278.5	41.98	12.313 H_2 found	99.78 % H_2
Exploded	151.4	23.51	12.34 gas added	

Same Gas.

Air	166.3	25.68	14.07 H_2O formed	
Gas added	231.0	35.08	9.38 H_2 found	99.79 % H_2
Exploded	134.3	21.01	9.40 gas added	

These analyses show that the air was not quite expelled from the generating flask before the gas was collected.

This apparatus is especially useful where it is desired to obtain reliable results in a comparatively short time. As before mentioned, it was designed more especially for the analysis of illuminating gas. It will be noticed that the explosion pipette of this apparatus is nearly the same as the Cavendish eudiometer; the chief difference being in the manner of introducing and expelling the gas.

Office of Gas Inspection, Boston, May 14, 1874.