XX.—*On the use of Gas as Fuel in Organic Analysis.*

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The extensive and daily increasing application of gas as a source of heat in the laboratory, naturally suggested the idea of using this convenient fuel likewise in organic analysis. A variety of arrangements have been proposed for this purpose, but not one of them has as yet come into general use. The difficulties which present themselves are, in the first place, the production of a sufficiently high temperature; and secondly, the regulation of the heat according to the special requirement of the process. In order to accomplish the analysis at the temperature of an ordinary air-flame, it has been found necessary to abandon the simple process of Liebig, and to have recourse to the more complicated method of completing the combustion in an atmosphere of oxygen; on the other hand, in order to obtain a sufficient control over the temperature, an arrangement has been devised, which may appear intricate at the first glance, but which works with sufficient facility and precision, although it may hereafter probably be simplified.

The general disposition of the apparatus differs but little from the arrangement used in performing the combustion in oxygen-gas by means of spirits of wine, and is sufficiently intelligible by a glance at Fig. 1.* *A* is the combustion-furnace, which is supplied with gas by

* The wood engravings are taken from Liebig's "Handbook of Organic Analysis," edited by A. W. Hofmann. London, Walton and Maberly, 1853.

FIG. 1.

the tubes *a b c d*, communicating with two pairs of stopcocks, *e* and *f*; a combustion-tube, *g*, open at both extremities and supported in the

usual manner, communicates in front with the chloride-of-calcium-tube *B*, a potash-bulb-apparatus *C*, and a potash-tube *D*; at the back, the combustion-tube communicates by means of a system of desiccators, consisting of a potash-tube *E*, a sulphuric-acid-tube *F*, and a sulphuric-acid-bottle *G*, with the gas-holders *H* and *I*, containing respectively oxygen and atmospheric air. The bottle *G* serves, moreover, as a bubble-gauge for measuring the rapidity of the current.

The furnace consists of three separate compartments of strong iron plate *h i* and *k*, which are supported by a stout iron stand *l m*. The arrangement of these compartments requires a few explanations; their construction is evident from Figs. 2 and 3; *h* and *i* are simply

FIG. 2.

FIG. 3.

rectangular iron boxes, open at the bottom and covered at the top, with wire-gauze, which are supplied by horizontal perforated gas-pipes (*n n*, Fig. 2). The last compartment *k* (Fig. 2) has a somewhat more complicated construction. It differs from the two others by being subdivided into four smaller chambers by the three diaphragms, *o*, *p* and *q*, of iron plate, the gas being supplied by

two pipes instead of one. The lower pipe *r r* resembles in every respect the gas-pipe *n n* of the other compartments. The upper pipe *s s*, on the other hand, supplies the gas to two rows of vertical fine tubes, somewhat similar to those used in Leslie's gas-burner, the extremities of which project through the wire-gauze cover of the compartment (Fig. 3). Both pipes are provided with air-tight pistons, which enable the operator to manage the heat produced by this part of the apparatus according to the requirements of the analysis. Fig. 3 shows the manner in which the frame *x* which supports the combustion-tube is fitted over the wire-gauze cover of the case. It is provided with a series of holes *a*, allowing a current of air to enter, which bends the flame round the combustion-tube; this figure gives, moreover, a separate view of one of the side-pieces *y*, which by means of the tongues *z* are fastened upon the frame, in order to form a kind of dome, from which the heat is reflected downwards upon the combustion-tube.

The combustion is conducted in the following manner: A combustion-tube, open at both ends, and 2 or 3 inches longer than the combustion-furnace, is filled with a layer of oxide of copper, corresponding to the length of the two compartments *h* and *i*, placed into the furnace and connected at one end with the desiccators *E F*, &c., the other end being open at the time. The tube is then heated by the air-flame of the three compartments, and a slow current of dry air forced through it by means of the gas-holder *I*, for about five minutes, which removes every trace of moisture. The current being interrupted, the front end is corked, and the supply of gas so far diminished in compartments *h* and *i* that a mere sheet of flame remains upon the wire-gauze, whilst it is entirely turned off from the compartment *k*. As soon as the tube has sufficiently cooled, the front end is connected with the chloride-of-calcium-tube *B*, the potash-bulbs *C*, and the potash-tube *D*, and a little platinum boat containing the substance to be analyzed, introduced at the other extremity, and pushed in about two-thirds of the division *k*. All joints being well secured, the gas is again fully supplied to compartments *h* and *i*, and the layer of oxide of copper corresponding to the length of these divisions heated to a dull redness. The distillation of the "substance" may now be commenced. For this purpose, the gas is admitted, by means of the flexible tube *d*, to the lower gas-pipe *r r*, and the piston *u* pushed so far from the inlet as to supply the gas to the chamber *o l* only, whilst it is excluded from the other chambers, Fig. 2. In this manner, an air-flame is obtained which keeps the posterior extremity

of the combustion-tube at a dull red heat. A small quantity of gas is next admitted to the upper gas-pipe *s s*, by means of the flexible tube *c*, the piston *r* being drawn so far from the inlet as to confine the gas to a very few of the little tubes, at the extremity of which mere points of flame are thus produced. The distillation now proceeds with perfect regularity, the slow air-current, which must be kept up all the while, as well as the high temperature of the posterior portion of the tube, driving all the products of combustion towards the red-hot oxide of copper, which effects their perfect combustion. The piston *v* is gradually drawn out, so as to bring all the jets into play, and the supply of gas to the jets increased as far as possible without producing too smoky a flame. As soon as the temperature of the jets is no longer capable of expelling volatile matter from the substance, the piston *u* of the lower tube is gradually pushed in, and the gas supplied to all the chambers of compartment *k*, until the whole of the combustion-tube is exposed to an uniform air-flame throughout its whole length. A new quantity of matter is thus expelled, but unless the substance under examination be volatile, a large quantity of carbon is left in the boat.

At this period the air-current is replaced by a slow stream of oxygen from the gas-holder *H*, the arrival of which in the combustion-tube is marked by a brilliant phenomenon of incandescence. After a few minutes, the boat is left clean, and the combustion is terminated. The current is, however, kept up for a very short time longer, and stopped only when pure oxygen may be traced at the mouth of the potash-tube by means of an ignited splint of wood.

By this time the products of combustion are perfectly swept from the tube, and lodged in their receptacles; moreover, the copper is completely re-oxidized; the flame is now gradually lowered, and the gas at last entirely turned off; and if the combustion-tube, after the detachment of the chloride-of-calcium-tube, &c., and the removal of the empty platinum boat be carefully corked, the whole arrangement is ready for another combustion, in which the operation of drying, of course, becomes unnecessary.

Liquids may be likewise burned with the apparatus; in fact, on account of the facility with which the temperature of the compartment *k* can be managed, this mode of proceeding is particularly adapted to liquids. Even very volatile liquids, such as ether, &c., may be safely burned; in this case the combustion can be completed without any oxygen, and no fear of an explosion need be entertained.

In the case of substances containing nitrogen, the copper turnings

perfectly dried in a current of hydrogen, must be placed into the combustion-tube, *after* the preliminary desiccation, and the current of air must be very slow; it is, moreover, advisable to place some copper-turnings behind the boat with the substance, over the posterior compartment; the copper is thus heated to redness, and deprives the air-current of the greater part of its oxygen.

The first putting together of this apparatus requires considerable time, and the securing of so many joints is attended with some difficulty; but once fitted up, it enables the operator to analyze with great dispatch, scarcely more than an hour being necessary for making a complete combustion. The arrangement is particularly useful when a considerable number of analyses have to be made. There are, moreover, several additional advantages which deserve to be noticed. The determinations, both of carbon and hydrogen, are very exact, especially the latter, all sources of accidental moisture being excluded. On this account it is possible to reduce the amount of substance used in the analysis to a minimum, not more than from 150 to 200 milligrs. being required. The possibility of determining the amount of ash or the quantity of silver, platinum or barium, &c., in a substance, together with the carbon and hydrogen, is likewise frequently a very great convenience. The number of accidents is, moreover, much smaller. The combustion-tubes very rarely crack if the application of heat be sufficiently gradual. For this purpose it has been found convenient to light the gas in the commencement, below the wire-gauze, until the furnace has become hot, then to turn it off and to light it above the wire-gauze. But if an accident actually take place with a combustion-tube, it almost invariably occurs in the preliminary heating of the oxide of copper, and in such cases the "substance" is perfectly safe, and has only to be shifted to another tube. The combustion-tubes usually stand six or eight combustions, but frequently as many as ten or twelve may be made with the same tube, so that there is also a considerable saving in expense.

The only inconvenience incident to the apparatus is, that it is liable to get out of order. The wire-gauze especially has to be often renewed, and on this account a construction has been selected, which renders it possible to replace the wire-gauze in the laboratory, without sending the furnace to a gas-fitter.

The furnace given in the above illustrations is composed of three distinct divisions, which may be separately fixed upon the support. By this provision the apparatus becomes adapted to a variety of operations, especially to nitrogen-determinations, which require a shorter tube. This separation, however, is not absolutely necessary,

and for the special purpose of the carbon-, and hydrogen-determinations a furnace made in one piece answers equally well.

In conclusion, the results of several analyses performed by means of the above apparatus may be quoted.

Combustion of sugar :

I.	0.155	gram. of sugar gave
	0.240	„ carbonic acid, and
	0.092	„ water.
II.	0.3035	„ sugar gave
	0.4685	„ carbonic acid, and
	0.1765	„ water.
III.	0.226	„ sugar gave
	0.3493	„ carbonic acid, and
	0.1319	„ of water.

	I.	II.	III.	$C_{12}H_{11}O_{11}$
Carbon . .	42.16	42.09	42.17	42.10
Hydrogen . .	6.59	6.46	6.48	6.43

Combustion of benzoic acid :

	0.1695	gram. of benzoic acid gave
	0.4275	„ carbonic acid, and
	0.077	„ water.

			$C_{14}H_6O_4$
Carbon . . .	68.78	. .	68.85
Hydrogen . .	5.04	. .	4.91

Combustion of oil of turpentine :

	0.1185	gram. of oil of turpentine gave
	0.3842	„ carbonic acid, and
	0.1240	„ water.

			$C_{10}H_8$
Carbon . . .	88.42	. .	88.23
Hydrogen . .	11.62	. .	11.77

Combustion of ether :

	0.1561	gram. of ether gave
	0.3695	„ carbonic acid, and
	0.1915	„ water.

			$C_8H_{10}O_2$
Carbon . . .	64.55	. .	64.86
Hydrogen . .	13.62	. .	13.51

The preceding analyses were made by Mr. Charles Tookey for the especial purpose of testing the practicability of the gas-furnace. The following numbers are quoted from a paper shortly to be published by Messrs. Reginald Morley and John Abel :

Combustion of diethyltoluidine :

0.1495 grm. of diethyltoluidine gave			
0.4435	„	carbonic acid, and	
0.1410	„	water.	
			$C_{22}H_{17}N$
Carbon	.	80.90	80.98
Hydrogen	.	10.47	10.43

Combustion of chloride of triethylo-toluummonium and bichloride of platinum :

0.530 grm. of salt gave			
0.760	„	carbonic acid,	
0.269	„	water, and	
0.131	„	of platinum.	
			$C_{26}H_{22}NCl, Pt Cl_2$
Carbon	.	39.10	39.24
Hydrogen	.	5.63	5.53
Platinum	.	24.71	24.90

An analysis of iodide of tetramethylammonium, likewise made by Mr. Tookey, gave the following results :

0.4138 grm. of iodide gave			
0.3626	„	carbonic acid, and	
0.2237	„	water.	
			$C_8H_{12}NI$
Carbon	.	23.89	23.89
Hydrogen	.	6.00	5.97