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Chromatographic Separation of Argon and Oxygen using Molecular Sieve

THE chromatographic separation of argon and oxygen is difficult, and existing methods depend on one of the following techniques:

(1) Separation on a molecular sieve 5 Å column at 18° C (ref. 1) and at −80° C (refs. 2–4).

(2) Chromatographing argon and oxygen as a composite peak on molecular sieve 5 Å at 100° C; removal of the oxygen with a reducing agent allows the argon peak only to be developed[5-7].

(3) Separation of argon and oxygen on a column of firebrick loaded with 8 per cent by weight of blood at 20° C (refs. 8 and 9).

It was noticed[10] that after a molecular sieve column was heated above the normal reactivation temperature of 300° C a partial separation of argon and oxygen resulted at a column temperature of 25° C. The operating parameters for optimum separation of argon and oxygen and the constancy of resolution with time have been investigated at this Laboratory using a molecular sieve 5 Å column. A helium ionization chromatograph[11] was used for this work.

The chromatographic column (5 ft. × 3/16 in. internal diameter stainless steel tube) was packed with Linde molecular sieve 5 Å (calcium alumino-silicate, 30–60 mesh) which had been previously heated to 400° C in air. The column was coupled to a helium ionization chromatograph and the molecular sieve heated to a minimum of 400° C for 24 h with a pure helium purge. Higher activating temperatures (for example, 500° C) do not appear to increase the efficiency of separation and the use of normal cylinder helium (no purification) does not give the required degree of reactivation.

A column treated in the foregoing manner gave the following relative retention times:

Hydrogen	Argon	Oxygen	Nitrogen	Methane
1·00	1·57	1·89	2·89	4·07

for a column temperature of 75° C and a helium flow rate of 67 ml./min. It was found that slight deterioration in resolution occurred over a 5-day period. This deterioration produced an increase in the argon retention time and a decrease in the oxygen retention time. Reactivation of the column at 400° C for 16 h gave the original retention times.

The effect of 'swamping' a small peak by a large amount of one component was found to be negligible for the range of volumes tested. A return to baseline between the argon and oxygen peaks was found for amounts of 20 μml. argon and 200 μml. oxygen and for 200 μml. argon and 19 μml. oxygen.

The sensitivity for both argon and oxygen was of the order of 1 μml. where the sensitivity is defined as five times the noise-level. A detector voltage of 500 V was used.

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Microwave Stimulation of Chemical Reactions

DURING an investigation of the effects of microwave energy on organic reactions, a novel application of such radiation has been disclosed. One well-known difficulty in the examination of solid-state pyrolytic reactions is that, perforce, for each particle, the products move from the site of their production against a positive temperature gradient. For any sizable particle, therefore, the pyrolysate is subject to further decomposition and, in many cases, it is impossible to isolate the primary products.

It has now been found that certain organic solids, notably coal and some similar substances, are effectively decomposed by microwaves and that the nature of the cracked product is different from that produced by purely thermal means. The reason for this difference is, I believe, because the microwaves decompose the material from the 'inside' outwards.

Small pieces of gas coal, about 1 c.c., are more or less instantaneously decomposed into a heavy tar precursor and a soft carbonaceous residue.

A Philips 7292 magnetron has been used and our equipment is a development of that of McTaggart[1]. The solids (1–3 g) are placed across the field in a wave-guide, within silica tubes, through which flows a slow current of nitrogen; the pressure is atmospheric. In the case of a typical gas coal, on switching on the anode current, within 2 sec heavy brown vapours were evolved which 'flowed' down the exit tube. The fog could not be effectively condensed by a liquid air trap but was collected by a packed plug of cotton wool. When allowed to deposit on surfaces, the fog coalesced to an orange varnish-like film, whereas, 'upstream' from the sample, the interior of the containing tube was covered for a short distance with a fine brown powder which, under the microscope, appeared as resinous particles. Immediately adjacent to the sample, the wall of the tube was covered with a carbon mirror, the inner surface of which was coated with fine carbon black. The residual coke was soft and friable. We have been unsuccessful, so far, in isolating the more conventional liquid products, such as are contained in 'light oil', etc.

The duration of the complete decomposition is so short (about 15 sec) that a conventional thermocouple placed outside the walls of the reaction tube does not have time to respond, although, if the power (700 W) be left on for 20–30 sec, the coke is raised to incandescence. Presumably, at lower power, uniform slow heating can be achieved.

With all coals, the decomposition was immediate and complete. With oil shales, there was often an induction period before reaction, whereas essentially aliphatic substances, such as torbanites, appeared transparent.