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FUEL RESEARCH INSTITUTE OF SOUTH AFRICA.

TECHNICAL MEMORANDUM NO. 23 OF 1967.

INTERIM REPORT ON THE DEVELOPMENT OF A NEW METHOD FOR THE DETERMINATION OF THE METHANE CONTENT OF COAL.

by

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INTRODUCTION:

The conventional method used for the determination of the methane content of coal consists of three steps, viz.

(i) procuring a suitable sample in the colliery,
(ii) extraction of the gas from the coal and
(iii) collection, measurement and analysis of the gas.

In this report an account is given of a new technique developed at the Fuel Research Institute whereby the methane evolved from a coal sample is automatically measured and analysed.

The method utilizes a gas detector of high sensitivity to monitor the methane content of a carrier gas that is used to sweep methane from a mill while the sample is being crushed.

An automatic sampling valve in the carrier gas stream feeds samples of the sweeping air from the mill into the detector at preselected intervals. The detector output is displayed on a recorder chart in the form of a series of peaks representing the methane content of the individual samples. The total methane content is obtained from the peaks by a simple calculation.

Apart from the quantitative measurement of methane the method also provides information on the rate of evolution of methane.

The method eliminates the collection, volumetric measurement and separate analysis of the gas.

The equipment is inexpensive and can easily be adapted for field use.

General...../

GENERAL DESCRIPTION OF THE APPARATUS.

A diagram of the apparatus is shown in Figure 1.

Dry compressed air is passed at a constant flow rate into the reference cell of a katharometer (thermal conductivity detector) and then, via a sampling valve into the measuring cell of the detector.

Air from the same source and at the same flow rate is passed through the mill in which the coal is being crushed, and into the sampling valve.

The valve is operated automatically in such a way that the air from the mill is alternately passed through the sampling loop or straight through into the atmosphere. The sample cut off in the sample loop is swept out by the pure air emerging from the reference cell of the detector into the measuring cell. Any methane present in the air sample causes an imbalance in the Wheatstone bridge (of which the filaments of the detector are two arms). The out-of-balance voltage is fed into a potentiometric recorder where it appears as a peak on the chart.

The sampling and analysis intervals of the sampling valve are adjustable by a bi-metallic time switch.

THE DETECTOR AND CARRIER GAS.

The choice of a katharometer as a detector was determined by the requirement that only methane evolved from the coal was to be measured.

Although nitrogen and oxygen and carbon dioxide also occur in coal they have no significance as far as explosion hazards are concerned.

By using air as a carrier gas the evolution of nitrogen and oxygen from a coal sample should not interfere unduly with the determination of the combustible gases. Furthermore, the air initially present in the extraction system need not be taken account of.

Carbon dioxide and water vapour, however, must be removed from the carrier gas. This is accomplished by inserting suitable absorbents in the gas line.

Hydrogen also occurs in coal but in such small quantities that no serious error will be involved by not removing it from the gas extracted from coal samples.

The...../

The difference in the thermal conductivities of air (5.83) and methane (7.14 cal/cm.sec.degree C x 10^5) is large enough to provide sufficient sensitivity for quantitative determination of methane by means of a katharometric detector.

THE SAMPLING VALVE.

Attempts to measure the amount of methane evolved by the sample by feeding the total gas stream directly into the detector proved unsuccessful. This is due to the variation in the amount of methane extracted during the grinding process.

During the period of maximum evolution overload of the detector occurs while in the initial and final phases low concentrations must be detected.

Since the detection system has a high sensitivity it seemed that intermittent sampling offered the best solution to the problem. Sampling of the carrier gas also has the advantage that disturbances in the gas flow rate caused by the milling operations are eliminated.

In order to facilitate the evaluation of the recorder trace it is essential to sample the carrier gas at regular intervals. This was accomplished by automating the manual gas sampling valve of a commercial gaschromatograph. The lever of the valve was connected to an air piston controlled by solenoid valves. A diagram of the device is shown in Figure 2.

A discarded aircraft hydraulic pump operated by about 10 lb/inch^2 air pressure was found satisfactory for moving the lever of the valve. The backward and forward movement of the pump piston was controlled by an adjustable bi-metallic time switch connected to the solenoid valves.

Sample loops of different sizes can be fixed to the sampling valve. A sample size of 2 ml. was found to be suitable for the amount of methane extracted from about 50 gm of coal.

EVALUATION OF THE RECORDER DATA.

A typical recorder chart showing the peaks obtained when doing a methane content determination is shown in Figure 3.

The total amount of methane evolved by a sample is represented by the area under the curve obtained by drawing a smooth curve through the apices of the individual peaks.

The...../

The area under such a curve can be determined by the following methods.

(1) Planimetry.

(2) Cutting and weighing of the paper.

(3) Electronic digital integration of individual peak areas and calculation of the total area (A_t) by means of the following equation.

$$\sum A_i \left(\frac{a+b}{b}\right) = A_t,$$

where $\sum A_i$ is the sum of the printer values of the individual peaks, a is the length of the base of a peak and b is the distance between the peaks at the base line (see Figure 3). The timing accuracy of the bi-metallic switch is sufficiently reliable to ensure constancy of a and b, respectively, for all the peaks.

(4) Electrochemical Integrator.*

The precision of area measurement of the individual methods, expressed in terms of standard deviations are:**

1. Planimetry	4.0%
2. Cut and weigh peaks	1.7%
3. Electronic Integration	0.4%
4. Solion Integrator	5.0%

Although the precision of the electronic integrator exceeds those of the other methods the uncertainties involved in other aspects of the measurement of the methane content of a coal sample annul the advantage of such high precision.

The cut and weigh method was therefore generally adopted for this work.

The electrochemical integrator is especially suitable for large peaks such as are obtained in this method. The device also produces an integration value directly on a dial. Its only disadvantage is that its precision is only 5%.

Calibration...../

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* Marketed under the trade name "Solion Integrator".

** Aerograph Corporation Information Circular.

CALIBRATION.

Individual peak areas represent the methane content of the air sample passed into the detector at that time.

To establish the relationship between peak area and methane concentration, calibrations are obtained by feeding a known quantity of methane into the carrier stream.

The conditions affecting the sensitivity of the detector such as carrier gas flow rate and filament current are kept constant during calibration and measurements.

THE GRINDING DEVICE.

In previous work the suitability of a horizontal oscillatory mill for grinding coal in a closed system was demonstrated (see F.R.I. Technical Memorandum No. 1 of 1965).

In the present investigation a similar device was employed except that the lid of the mill was provided with two air-tight connections through which the carrier gas is passed.

CONCLUSION.

The apparatus described has distinct advantages over previously described methods for the determination of the methane content of coal.

The sensitivity of the detecting system permits the use of small samples of coal taken from the inside of large samples brought from the colliery. This procedure reduces the sampling error involved considerably because that portion of the sample subjected to testing is less exposed than the outer parts of the sample.

Although this aspect of the procedure has not yet been fully investigated it seems likely that the use of gas-tight containers for storing the samples can be dispensed with. The use of gas-tight containers has the limitation that lumps no larger than the container mouth, which is only about 3 inches can be used as samples. The loss of methane from such small lumps can be expected to be much more than in the case of samples taken from the inside, of say, 12 inch blocks immediately before testing.

Another advantage of the new method is that the extraction and analysis of the gas are undertaken simultaneously,

resulting...../

resulting in a considerable saving in testing time. The earlier methods required first the extraction, then volumetric measurement and finally the analysis of the gas. This procedure requires at least two hours compared with about one half hour with the new technique.

The new method also affords a means of studying the kinetics of the methane desorption rate and how it is influenced by grinding and temperature.

PRETORIA.
12th July, 1967.

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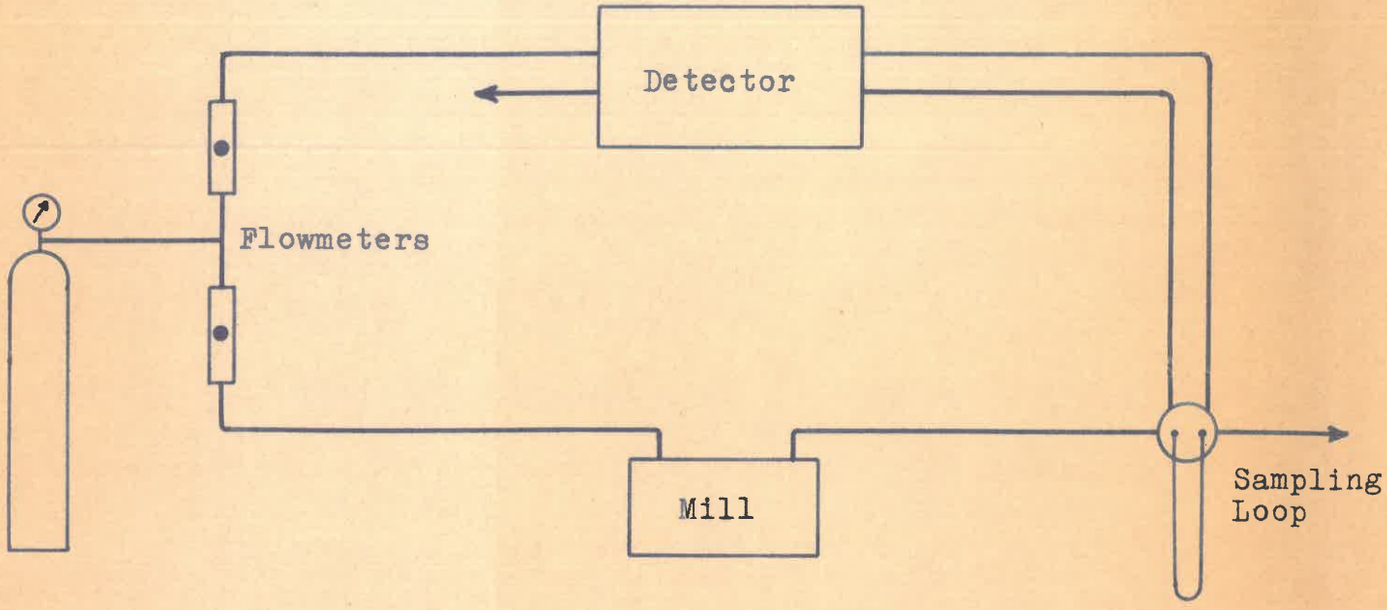

FIGURE 1

Flow Diagram of Apparatus

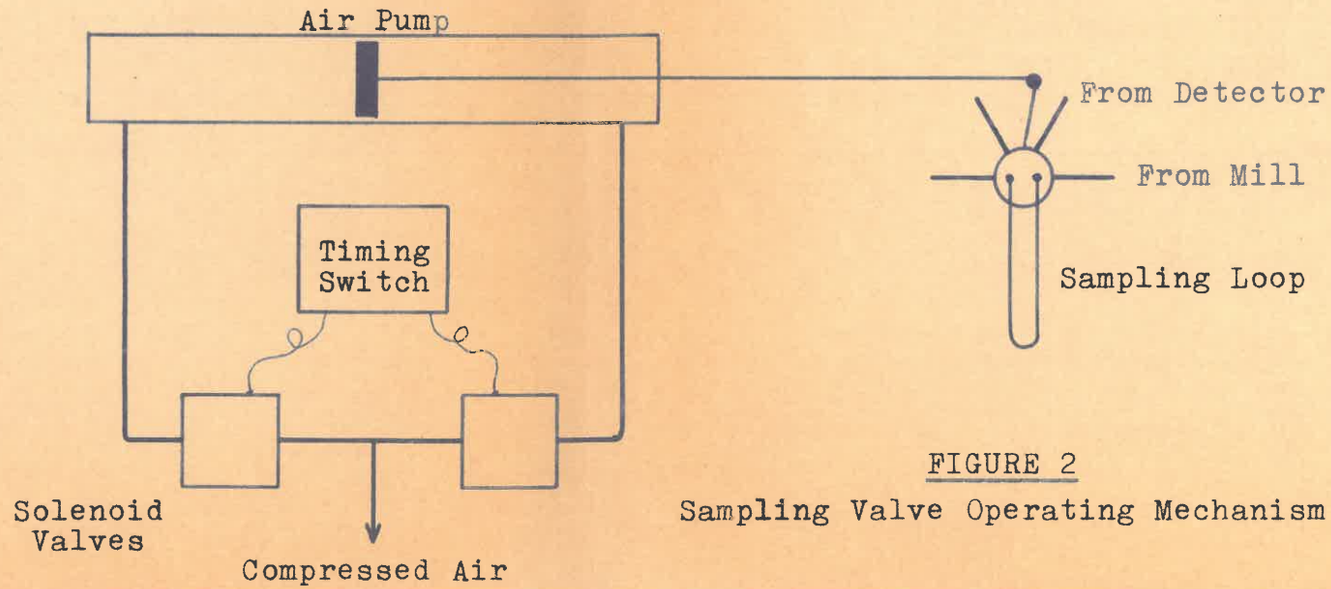

FIGURE 2

Sampling Valve Operating Mechanism

FIGURE 2

Sample Weight	50 gm.
Detector Flow Rate	40 ml./min.
Mill Flow Rate	40 ml./min.
Bridge Sensitivity	5.0
Recorder Sensitivity	1 mV.
Chart Speed	20 m.m./min.
Start of run at A