Rapid Analysis of Simple Gas Mixtures
Schnellanalyse einfacher Gasmischungen
Analyse rapide de mélanges gazeux simples

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Summary: This paper describes the use of readily prepared columns and a microkatharometer for the rapid analysis of simple gas mixtures. The sample size used is 2 microlitres and the linear response from the detector for all gases investigated makes calibration easy. The limit of detection for oxygen is about 0.1%.

Introduction
The gas industry makes many routine analysis of gas mixtures containing a small number of components. This paper is concerned with increasing the speed of analyses for such simple gas mixtures.

Wilhite [1] demonstrated the use of refined micro-packed columns for the rapid analysis of gas mixtures. His columns were of 0.25 mm ID and contained column packing of between 25 and 28 microns in size.
Packed capillary columns [2] and support coated open-tube (Scot) columns [3] have also produced useful results in rapid analysis.

Small samples are common to these techniques. For detection of peaks in small samples by thermal conductivity it is necessary to use sensitive low volume micro katharometers.

This paper describes a method for the rapid separation of simple gas mixtures by means of short columns prepared from 1 mm ID tubing and containing conventional chromatographic stationary phases.

Experimental
A low volume sample valve coupled with a micro-katharometer was used in this work; both were made by Servomex Controls Ltd. The sample valve, type SV202, could hold between 2 and 40 microlitres of sample. A sample volume of 2 microlitres was generally used.

The micro katharometer (type MK158) output was amplified by a variable gain amplifier and recorded by an ultraviolet galvanometer recorder.

Columns were prepared from 1 mm ID stainless steel tubing and plugged with cotton or silica wool. The column packing materials used are indicated in Fig. 1–3.
The gain setting of the amplifier was used to attenuate the peaks. Since manual attenuation by means of the gain control is more or less impossible during an analysis, an automatic attenuator was also used in conjunction with the amplifier. It was constructed from Norbit integrated circuit modules (MEL Equipment Co. Ltd) and consisted of a series of logic modules controlling timing circuits. The sample injection started the logic sequence; then the timers sequentially switched the amplifier output through various resistances in the matching network of the galvanometer.

Multiple galvanometers, as used to record mass spectra, might have been a simpler method of attenuation. The chromatograph was calibrated by means of gas mixtures prepared in a gas burette. An exponential – dilution flask [4] was used for the study of the linearity and the limits of detection of the system. It consisted of a beaker fitted with a rubber bung provided with inlet and outlet tubes for gas flow. The contents of the beaker were stirred by a large vane rotated by a magnetic stirrer. Sample gas was used to purge the beaker; then the inlet line was switched to hydrogen. The effluent from the beaker passed through the sample valve and samples were introduced into the chromatograph at regular intervals during the dilution.

Results and Discussions

Typical chromatograms are shown in figures 1, 2 and 3 together with the respective chromatographic conditions. Figure 3 shows also the effect of the automatic attenuator. The sample was the same as used for figure 2 but the carbon dioxide and ethane peaks were attenuated by a smaller resistance than were the carbon monoxide and methane peaks.

The response of the chromatograph is linear with respect to peak height for all concentrations down to the limits of detection. The linearity is shown in figure 4 which shows the results of an exponential dilution of air with hydrogen carrier gas. The limit of detection of oxygen under these conditions was about 0.1%; this limit could be lowered by using larger samples.