



Analysis of gaseous sulfur compounds by process gas chromatography  
by Edward I-Sen Wan

A Thesis submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree of  
MASTER OF SCIENCE in Chemical Engineering  
Montana State University  
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Abstract:

A process gas chromatograph for the quantitative determination of gaseous sulfur compounds has been developed and successfully tested on the prepared samples. The instrument is an improved version of the instrument reported by Applebury (4). The sensitivity and precision are increased by replacing the column packing with PORAPAK Q-S material and increasing the titration solution concentration by twenty percent.

A detailed study of the variation of peak size, with sample pumping time, cell adjustment and titrating solution concentration is reported.

The calibration data of hydrogen sulfide, sulfur dioxide and methyl mercaptan are presented at different operating conditions. The study of higher order sulfur compounds, as well as the effects of temperature, carrier gas flow, and steam condensation is also reported.

The instrument can analyze wide concentration ranges of gaseous sulfur compounds (from 0.1 ppm to 1000 ppm by volume), and the analysis may be performed by remote-control at ten minute intervals.

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BY PROCESS GAS CHROMATOGRAPHY

by

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## ABSTRACT

A process gas chromatograph for the quantitative determination of gaseous sulfur compounds has been developed and successfully tested on the prepared samples

The instrument is an improved version of the instrument reported by Applebury (4). The sensitivity and precision are increased by replacing the column packing with PORAPAK Q-S material and increasing the titration solution concentration by twenty percent.

A detailed study of the variation of peak size, with sample pumping time, cell adjustment and titrating solution concentration is reported.

The calibration data of hydrogen sulfide, sulfur dioxide and methyl mercaptan are presented at different operating conditions. The study of higher order sulfur compounds, as well as the effects of temperature, carrier gas flow, and steam condensation is also reported.

The instrument can analyze wide concentration ranges of gaseous sulfur compounds (from 0.1 ppm to 1000 ppm by volume), and the analysis may be performed by remote-control at ten minute intervals.

## I. INTRODUCTION.

The Kraft pulp mill has been reported to be a significant contributor to air pollution, and intensive studies are in progress to minimize pollutants from this source. These studies have necessitated the development of analytical techniques capable of obtaining definitive information on the compositions of mill exhaust.

Many gas chromatographic methods for determining gaseous sulfur compounds have been reported in the literature (1, 2, 3), but none of these methods have the capability of continuously monitoring pollutants from pulp mills.

The process gas chromatograph used in this work was originally constructed by Applebury (4), and the instrument described in this thesis is an improved version of that instrument. The sensitivity and precision were increased tenfold by several modifications. These are:

- (1) replacing the column packings with PORAPAK Q-S (100/120 mesh), from Van Waters Assoc. Inc.,
- (2) replacing all tubing and connections with teflon or stainless steel materials,
- (3) replacing the sample valve with a Wesco solenoid valve, model No.A973,
- (4) improving the sampling control box and sample injector,
- (5) increasing the KBr concentration in the titrating solution by twenty percent, and
- (6) increasing the carrier gas flow rate to 200 ml./min.



These modification provided the desired increased sensitivity without loss in resolution.

## II. EQUIPMENT AND EXPERIMENTAL PROCEDURES

### INSTRUMENTS

The process gas chromatograph consists of an analyzer, a control unit, and a data processing system.

#### A. Analyzer

The analyzer provides the analysis of the samples. It has a sensitivity of 0.1 ppm to 1 ppm depending on the individual sulfur compound (for a detailed description of the analyzer, the reader is referred to the thesis of Applebury (4)). The schematic diagram of the analyzer is shown in Figure 1.

The detector was a three section bromine coulometric microtitration cell. The position of the sensor electrode was changed so that the capillary tube was perpendicular to the platinum plate of the sensor electrode. The new position is shown in Figure 2. The column packing was replaced with PORAPAK Q-S (100/120 mesh).

#### B. Control Unit

A control unit governed by a programmed timer mechanism provided remote control of the analysis. The function switch in both the control panel and the microcoulometer permitted either manual or automatic analysis of the sample mixture. A detailed discussion of this unit was described in Applebury's thesis (4).

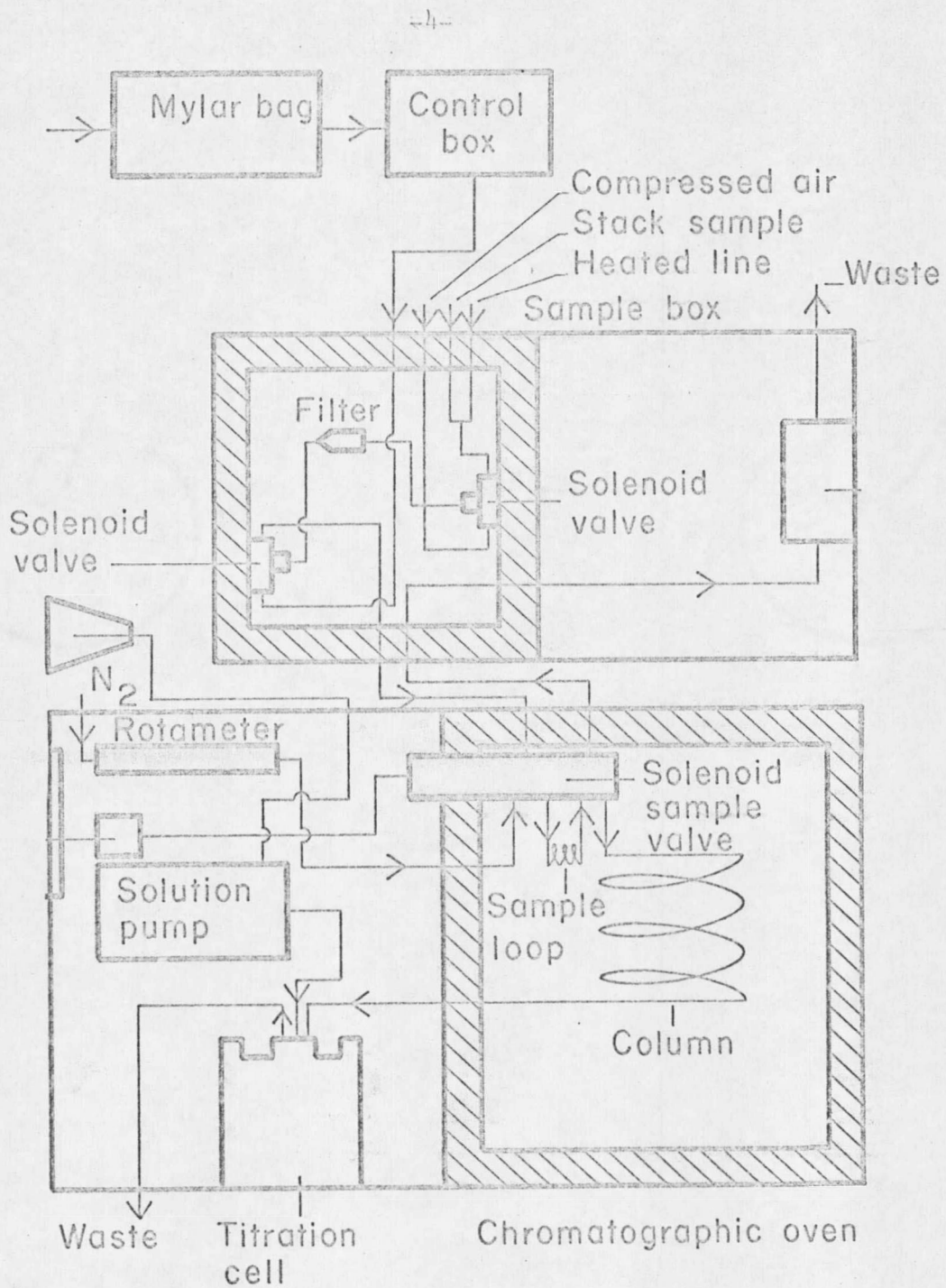


Figure 1. Analyzer































































