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AEC RESEARCH AND DEVELOPMENT REPORT

A DERIVATIVE COMPUTER FOR THERMOGRAVIMETRIC ANALYSIS

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A DERIVATIVE COMPUTER FOR THERMOGRAVIMETRIC ANALYSIS

by

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ABSTRACT

A derivative computer was designed for use with an Ainsworth thermogravimetric analysis system. The basic circuit, a simple differentiator using an operational amplifier, is very stable and accurate.

INTRODUCTION

Derivative thermoanalytical techniques have many applications.[1,2] Recording the first derivative of sample weight loss along with the weight curve and temperature during thermogravimetric analysis provides valuable information for sample analysis, particularly for complex samples with many small weight changes. The weight curve is often complex and hard to interpret by graphic means. Correlations between the observed temperature and the start of a chemical reaction in the sample can be precisely and easily obtained with the derivative curve. The first derivative is also useful for calculating the procedural energy of activation of the reactions. This report describes a sensitive derivative computer that shows the beginning of a weight change earlier than a weight chart.

PRINCIPLE OF OPERATION

The derivative computer is a simple differentiator circuit built with an operational amplifier. Signal integration, or filtering, was necessary to reduce the circuit bandwidth and to improve the output signal-to-noise ratio. The input signal to the differentiator is a very low frequency voltage proportional to sample weight and is derived from a single-turn infinite-resolution potentiometer mechanically coupled to the recording pen of a Bristol recorder. The Bristol recorder is part of a measuring bridge in the Ainsworth thermogravimetric analysis system and records the sample weight as a function of time.

CIRCUIT DESIGN

The instrument is shown in Figure 1. The circuit is shown in Figure 2. The potentiometer on the Bristol recorder of the Ainsworth system was connected to a stable power supply to give a 15-volt input change for a 10-milligram change in the sample. The desired full-scale output range of the derivative computer, based on typical weight charts from previous tests, is 2.5 to 20 micrograms per second. The input potentiometer derives a signal of 1.5 millivolts per microgram. Component values for the differentiator stage (Figure 2, A-1) were calculated by

$$e_i K_{max} = \frac{e_o}{R_2 C_1}$$

where

e_i = input voltage, millivolts per microgram

K_{max} = maximum range, micrograms per second

e_o = output voltage, volts

R_2 = resistance, ohms

C_1 = capacitance, farads

R_2 and C_1 were selected to make the circuit time response less than one minute. Capacitor C_1 must be of a low leakage type, such as polystyrene. The operational amplifier must be selected for low noise and low input leakage current.

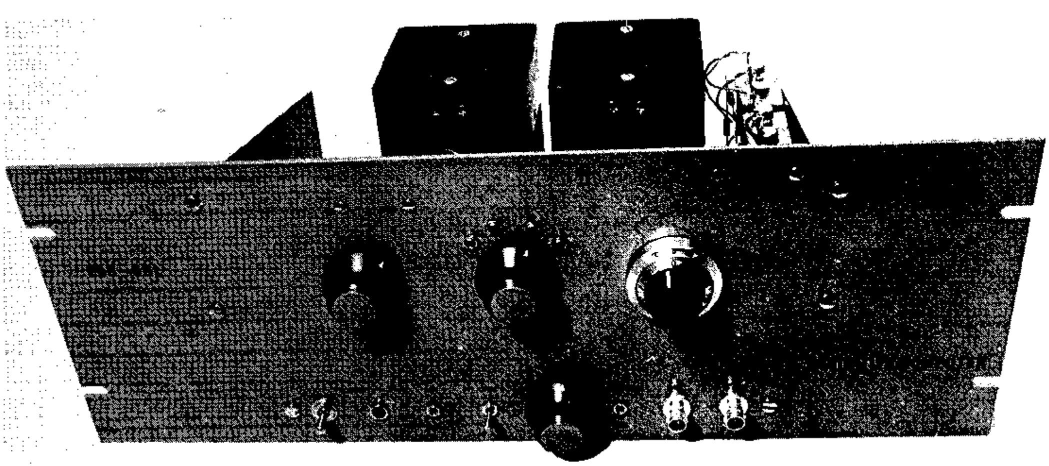

FIG. 1 DERIVATIVE COMPUTER INSTRUMENT

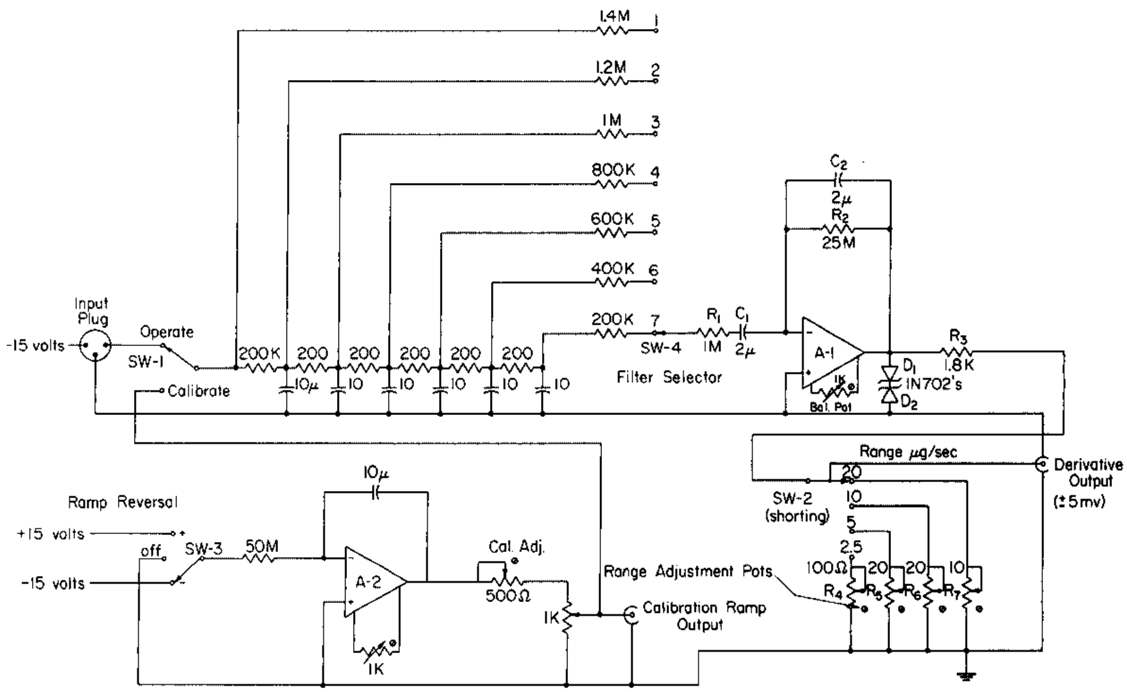

FIG. 2 CIRCUIT FOR DERIVATIVE COMPUTER

The differentiator output voltage is attenuated to 5 millivolts with a switch that provides four output ranges.

Input filtering was necessary to reduce system noise. The filter selection circuitry uses high precision resistors and low leakage capacitors for constant differentiator gain. The primary source of noise is the weight balance mechanism. The input filters naturally introduce circuit delays (Table I).

An integrator stage (Figure 2, A-2) gives a stable, linear variable ramp voltage for testing and calibrating.

Table I

Measured System Response to a Ramp Voltage Input

Input Filter Setting	Output Delay, sec	Time to 90% Final Value, min	Time to 95% Final Value, min
F-1	-	0.35	0.4
F-2	1.5	0.9	1.35
F-3	3	1.45	2
F-4	4	1.75	2.35
F-5	6	2	2.55
F-6	7	2.15	2.7

PERFORMANCE

Tests using the built-in ramp signal as the input voltage, and a "Servowriter II"* recorder are summarized in Table II.

The derivative computer described is a very accurate and stable instrument. The output signal-to-noise ratio for normal operating conditions can probably be improved greatly by deriving the input signal from the bridge measuring circuit of the Ainsworth system instead of from the potentiometer in the weight recorder. The instability, or "hunting," of the recorder is introduced as a noise signal to the differentiator. If a potentiometer is used, it must be an infinite resolution type for maximum signal-to-noise ratio.

Table II

Circuit Performance

Noise, input shorted	<.01 μg/sec
Time response	See Table I
Error due to -15 volt input signal[a]	0.025 μg/sec
Linearity of selected range	<1% full scale
Accuracy between ranges	1% full scale
8-hour stability, input shorted	<0.01 μg/sec

[a] Input voltage: 0 mg on recorder = 0 volts
10 mg on recorder = -15 volts

* Trademark of Texas Instruments.

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