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The Design, Construction, and Some Uses of an Automatic Recording Balance

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Introduction

Many chemical and physical reactions are accompanied by changes in weight of the reactants due to evaporation of liquids, evolution of gases, or adsorption of gases. Studies are made using data which show the manner in which the weight changes with time. Kinetic studies are made from a knowledge of the rate of loss or gain of a component. The drying of a precipitate to constant weight involves a loss in weight. Rates of evaporation of liquids from different types of surfaces involve changes of weight as a function of time.

Several instruments and methods are used to follow weight changes. Gas buret systems are used to measure the volume of a gaseous component which is adding to or subtracting from the weight of a reactant. The ordinary analytical balance is used to follow weight changes by making intermittent weighings. This latter method involves tedious operations and interruptions of the treatment of the reactants.

Effort has been put forth to devise instruments which will automatically weigh substances and record these weighings on some type of graph. Gregg and Wintle (1) devised a glass automatic electrical sorption balance. Gregg (2) later described a recording system for this type balance. Eyrand (3) reported a glass sorption balance of a somewhat different type. Duval (4) described a thermal balance that automatically records changes in weight on film.

Described in this paper is an instrument attached to a modified ordinary analytical balance which automatically records on graph paper changes in weight as a function of time.

Materials

This instrument consists of three basic units, the balance (Fig. 1-b), the electronic control unit (Fig. 1-c), and the recorder (Fig. 1-f). The materials for the construction of these units are standard and easily available with the exception of the recorder. The design is such, however, that many types of recorders could be used. Each basic unit will be described below.

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A. Balance

The balance (Fig. 1-b) is a slightly modified ordinary analytical balance from which the pan supports were removed and ¼ inch holes were bored through the bottom of the pans and balance case. Number 16 nichrome wire rods were suspended from hooks on the balance arms through these holes. A reaction vessel hangs from the left rod into some type of constant temperature gas bath as explained later. On the right rod is suspended a 1 x 6 cm. Alnico magnet sealed in a glass tube which hangs within the core of the

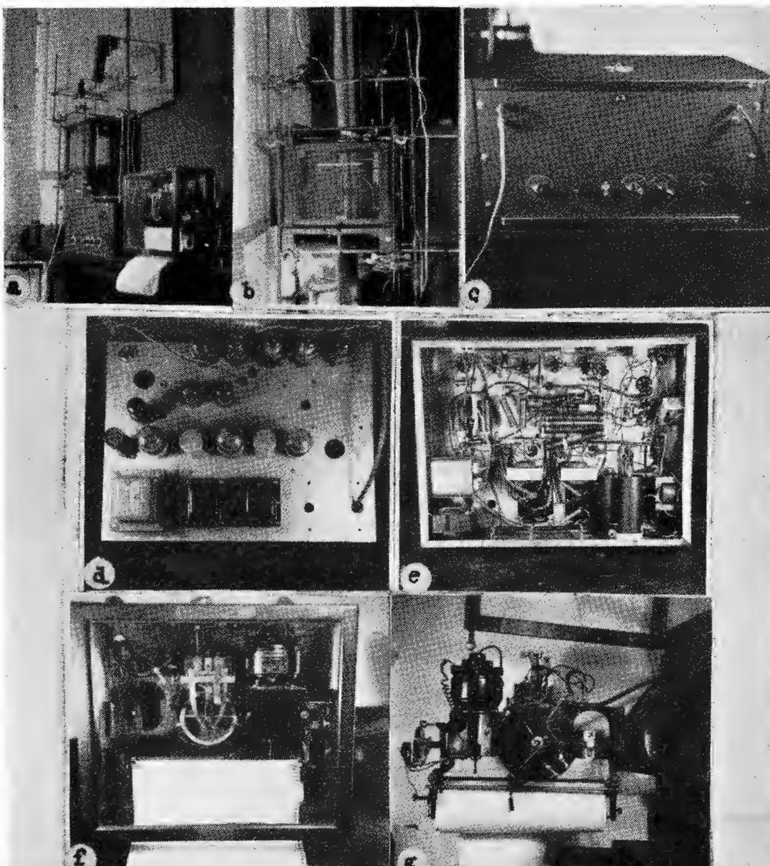


Figure 1. Automatic Recording Balance
- a. Balance in operation.
- b. Balance.
- c. Electronic control unit.
- d. Electronic chassis, upper view.
- e. Electronic chassis, bottom view.
- f. Recorder.
- g. Rear view of recorder showing drum potentiometer.

coils attached to the electronic control unit. The details of these hand-wound coils are shown in table 1.

Table 1

Details of Control Coil Windings

Coil	Length	Core	Wire Used	Diameter over Winding	Resistance
Control Coil	5 cm.	4 cm.	27 B.&S. enameled Cu.	5.5 cm.	Approx. 115 ohms
Damping Coil (Double Winding)	5 cm.	4 cm.	32 B.&S. enameled Cu.	6.5 cm.	Approx. 110 ohms (each winding)

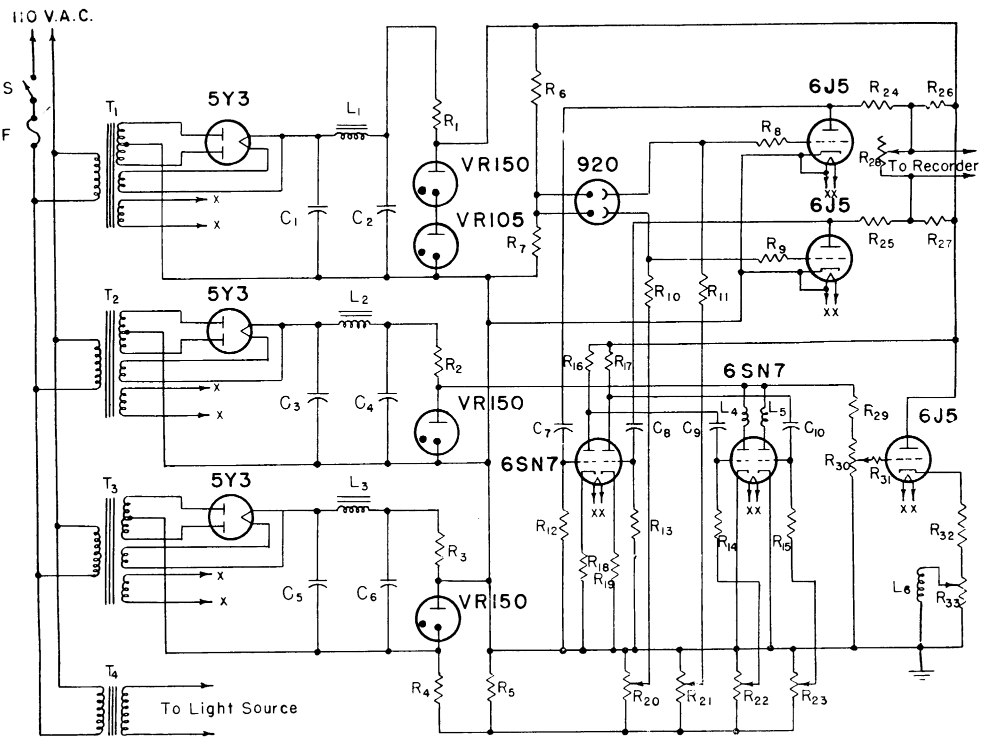


Fig. 2 Circuit Diagram

A small mirror (1 cm. x 1 cm.) is mounted exactly in the center of the balance beam. The mirror is masked with black paper to leave in its center a circle 2 mm. in diameter. A light beam is focused from an approximately 45° angle onto the mirror from a light source mounted above the balance case (Fig. 1-b). The light source is a 6 volt No. 82 bulb mounted in a housing. A lens in front of the bulb focuses the filament image onto the mirror. This image is then reflected through a reading glass lens onto a dual photocell mounted in a housing above the balance (Fig. 1-a & b).

B. Electronic Control Unit

The electronic control unit (Fig. 2) was designed to provide the maximum reliability and sensitivity consistent with low cost and ease of construction. Only parts which are readily available from standard radio supply houses are incorporated in the circuit. Where possible, conventional designs for power supplies, amplifiers, etc. are used. Since no high frequency circuits are employed, the construction of this instrument requires no great skill and the chassis layout is not critical. Ability to use a soldering iron and hand tools is about all that is required of the builder of this instrument.

For the most part, strict adherence to the specifications regarding electrical size of components (Table No. 2) is not necessary. A person with some knowledge of electronic circuits can easily modify the design to facilitate utilization of electronic parts that the builder may already have on hand. Modifications of the circuit will be necessary in the event that it is desired to use a recorder not of the old mechanical type described in this paper. An explanation of the various major parts of the circuit follows below:

Regulated Power Supplies

All of the d.c. power supplies are regulated by means of glow discharge, gas-filled voltage regulator tubes. This is the simplest type of regulation and is sufficient only if the input voltage is fairly constant. The writers found it expedient to obtain the 110 V. A.C. input from 220 volt line by means of a step-down transformer. This 220 volt source was not subject to the extreme voltage variations found in the 110 volt line. Providing the frequency of the source does not vary, a constant voltage transformer of the magnetic saturation type may be employed for preliminary stabilization of the a.c. input. The rectifier and filtering circuits used are of conventional design and have a nominal current carrying capacity of about 40 ma. The power supplies are all contained in the main chassis assembly.

Table 2

Electronic Parts List*

C_1, C_2, C_3, C_4, C_5, C_6—10 mfd. electrolytic, 450 V.
C_7, C_8—5 mfd. paper, 400 V.
C_9, C_{10}—0.1 mfd. paper, 400 V.

L_1, L_2, L_3—Filter choke, 16 hys., 50 m.a.
L_4, L_5—Damping coils.
L_6—Main control coil.

F—Fuse, 5 amp.

R_1—1500 ohms, 5 watt.
R_2, R_3—10,000 ohms, 10 watt.
R_4—50,000 ohms, 1 watt.
R_5—5,000 ohms, 1 watt.
R_6—160,000 ohms, ½ watt.

R_7—220,000 ohms, ½ watt.
R_8, R_9—4 megohns, ½ watt.
R_{10}, R_{11}, R_{12}, R_{13}, R_{14}, R_{15}—1 megohn, ½ watt.
R_{16}, R_{17}—25,000 ohm, 1 watt.
R_{18}, R_{19}—1500 ohm, 1 watt.

R_{20}, R_{21}, R_{22}, R_{23}—Potentiometer, linear taper, 500,000 ohms.
R_{24}, R_{25}—2500 ohm, 1 watt.
R_{26}, R_{27}—50 ohm, 1 watt.
R_{29}—Potentiometer, linear taper, 100 ohms.
R_{28}—100,000 ohms, ½ ohms, 1 watt.

R_{30}—Drum potentiometer, linear taper, linearity tolerance—0.5%, 10,000 ohms.
R_{31}—250,000 ohms, 1 watt.
R_{32}—10,000 ohms, 1 watt.
R_{33}—Potentiometer, linear taper, wire-wound, 500 ohms.

s—Swith, SPST., toggle.

T_1, T_3—Power transformer, 235-0-235 volts, rms., 40 ma.
T_2—Power transformer, 300-0-300 volts rms., 70 ma.
T_4—Filament transformer, 6.3 volts rms., 6 amp.

*Symbols refer to Circuit Diagram (Fig. 2).

Photocell and Photocell Amplifiers

The photocell and the first amplifier stages are contained in a separate housing made from a 4″ x 5″ x 3″ utility cabinet. This cabinet, complete with built-in chassis, may be obtained from almost any radio supply house.

The parts mounted in this cabinet are the 920 phototube, the two 6J5 amplifier tubes, resistors R_6, R_7, R_8, R_9, R_{10}, and R_{11}, and the potentiometers R_{20} and R_{21}.

An external mounting plate and hood for the phototube was constructed from light-weight sheet metal and fastened to the cabinet with self-tapping screws. The cabinet was fitted with a brass rod for mounting in an ordinary ring-stand clamp. This chassis is connected with the main chassis assembly by means of a seven-conductor shielded cable with an eight-contact multiwire cable connector mounted in the main chassis assembly.

The 920 phototube employed is a twin-unit gas filled type with maximum sensitivity in the range of 800 to 1000 mμ. This is the best spectral sensitivity for use when an ordinary incandescent bulb is the light source. The 6J5 triodes which constitute the first stage of amplification of the photocell current are mounted close to the phototube to provide short leads for the extremely small currents from the phototube. The apparent sensitivity of each half of the photocell can be varied independently by means of the potentiometers (R_{20} and R_{21}, Fig. 2) which control the grid bias voltage of the amplifier tubes.

The output of the photocell amplifiers is applied to the recorder terminals through a bridge circuit (R_{24-27}) and also to the damping circuit described below. A 100 ohm variable resistor (R_{28}) shunted across the recorder terminals provides for adjustment of the recorder response.

Cathode Follower

Attached to the shaft of the recorder pen mechanism is a 10,000 ohm precision linear potentiometer R_{30}. This potentiometer supplies to the cathode follower amplifier tube a grid voltage which varies with the motion of the recorder pen. The plate (and cathode) current of this tube is in direct and linear proportion to the grid voltage applied and hence is directly dependent on the position of the recorder pen. A portion of this current is fed to the main control coil L_6 thus supplying a magnetic field which is controlled in a linear manner by the recorder pen mechanism. This magnetic field surrounding the Alnico magnet suspended from the balance beam controls the position of the beam. The fraction of the total cathode follower current that is applied to the control coil may be varied by the 500 ohm potentiometer (R_{33}) in the cathode supply. This provides a means for adjusting the range (number of chart paper divisions per milligram) of the balance.

Damping Circuit

A time lag between a change in the magnetic field of the control coil and the response of the balance beam always exists due to inertia of the beam. This results in a continual over-correction whenever there is a change in weight of the sample. If no provision were made for this over correction, the balance would then remain in continual oscillation. The operation of the damping circuit incorporated in this instrument to eliminate this difficulty may be described as follows:

Consider the light beam from the mirror on the balance beam to be sweeping onto the top cathode of the 920 phototube in Fig. 2.

The rate at which this occurs depends upon the natural period of oscillation of the balance. As the illumination increases, the current through that half of the phototube increases and causes the following series of events:

1. The voltage drop across R_{11} resulting from the increased current through R_{11} makes the grid of the top 6J5 more positive.
2. Raising the grid potential increases the plate current and lowers the potential at the plate due to a larger IR drop in the resistors R_{24} and R_{26}.
3. This reduces the current through the coupling condenser C_7 and resistor R_{12} lowering the grid potential of that half of the first 6SN7 amplifier tube. No differentiation of the voltage across C_7 and R_{12} occurs because the time constant $R_{12} \times C_7$ (20 sec.) is long compared with the period of the balance.
4. The plate current in that half of the second 6SN7 decreases causing a reduction in the IR drop across R_{16}. The potential at the plate and hence at the top of the condenser C_9 increases.
5. C_9 and R_{14} constitute a simple differentiating circuit since the time constant $C_9 \times R_{14}$ is small (0.1 sec.) compared with the time required for the light beam to swing completely onto the phototube (i.e., the period of the balance). Thus as the voltage applied to C_9 increases an instantaneous voltage is produced across R_{14} which is proportional to the differential with respect to time of the voltage applied across C_9 and R_{14}.
6. This voltage across R_{14} makes the grid potential of that half of the 6SN7 more positive causing a current to flow in the damping coil L_4. The overall result is that the current flowing in L_4 is proportional to the rate at which the light beam is moving onto the phototube which is also the rate at which the balance beam is swinging. Since this tube is biased below cut-off by means of the damping control potentiometer, R_{22}, no current flows in the damping coil unless the illumination of the phototube is increasing. An exactly parallel operation occurs when the beam swings in the opposite direction. The current which flows in the damping coils in either case is such that the magnetic field which is set up tends to oppose the motion of the magnet, restoring the beam to the equilibrium position.

C. Recorder

The recorder (Fig. 1-f) is an old revamped Leeds and Northrop temperature recorder. A constant speed motor, the speed of which can be controlled by an adjustable governor, turns a roller for the chart paper. A cammed drive shaft operates a drum pen mechanism. The pen mechanism is controlled by a galvanometer which is deflected to and fro in response to swings of the balance beam.

Attached directly to the shaft of the pen mechanism is a 10,000 ohm linear drum potentiometer (Fig. 1-g) which turns with the pen mechanism a number of degrees proportional to the deflection of the galvanometer needle. This potentiometer varies the amount of current flowing in the control coils in such a sense as to tend to restore the balance to a null position.

As mentioned above other types of recorders that operate on varying current signals could be used with this balance. When no recorder is available it would be possible to construct one as described by Gregg (2).

Operation of Balance

The instrument operates on 110 volts A.C. Several controls are used in its operation. Two photocell potentiometers are mounted on the photocell housing (Fig. 1-b). These vary the sensitivity of the photocells and are set to zero the galvanometer needle of the recorder when the balance is at its zero point. Other controls are mounted on the panel of the electronic control unit. These will be described from left to right as shown in Figure 1-c. First is a sensitivity control which is used to determine the extent of deflection of the recorder galvanometer needle for a given deflection of the balance beam. Next is a fuse box, then the master switch for the electronic control unit and light source. The next two controls are used to adjust the amount of damping required. They operate in parallel, each controlling one damping circuit. At the right side of the panel is the range control which determines the number of lines on the chart paper that corresponds to a given change in weight.

After a warm up period, the balance beam is locked and the recorder galvanometer needle is zeroed by the photocell potentiometers. The balance beam is unlocked and tares are added to one balance pan until the recorder drives the pen to the desired position on the chart paper. The damping controls are turned clockwise in parallel until sufficient damping is observed. Once the proper damping is found very little further adjustment of these controls is needed. The range is found by setting the range control and observing the plot made when a given weight is added to one balance pan. Turning the range control in a clockwise direction decreases the range. The chart paper speed can be changed by a screw adjustment on the governor of the recorder motor.

Finally a weighed load is introduced into the reaction vessel and tared by an equal weight placed on the right pan of the balance. The instrument then automatically plots any changes in weight of the load as a function of time.

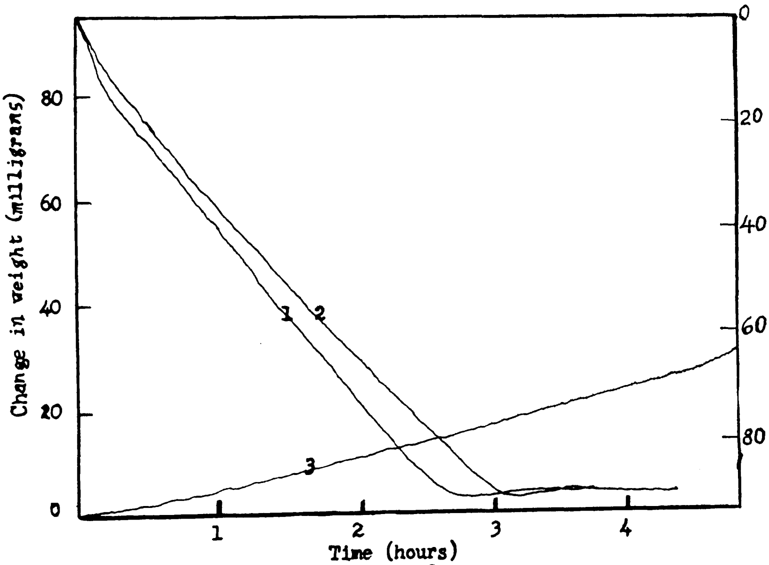
Accuracy, Sensitivity, and Range

The range of this instrument is adjustable by the range control from 100 milligrams to 25 milligrams per chart-paper width. This range could be further increased by a small modification in the electronic circuit. Changes in weight are recorded with an accuracy of 0.5%. The instrument is reproducible to ± 0.1 milligrams. Over extended periods of time the uncertainty is ± 0.2 milligrams. Although the electronic control unit contains voltage regulators, excessive fluctuations of line voltage can result in increased uncertainty. By using a 220 volt source, which is usually more constant, and a transformer for converting to 110 volts this uncertainty can be decreased.

Some Uses of the Balance

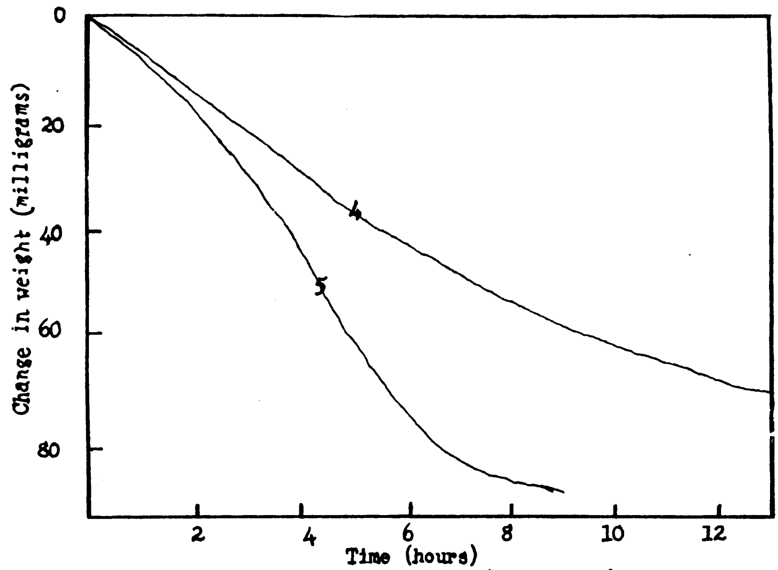
This instrument can be used in many ways for a variety of studies. It will plot changes in weight of most reactants that one might choose to place in the reaction vessel, provided their weights are no more than the load normally placed on an analytical balance. The nature of the reaction to be studied will determine the size, shape, and environment of the reaction vessel used.

In rate studies constant temperature is usually necessary. For studies at room temperature all that is required with this balance is proper shielding of the reaction vessel from air currents. For low temperature work one suspends the reaction vessel into a slightly larger metal tube surrounded by some freezing mixture. For higher temperatures a liquid constant temperature bath surrounds



Curve 1. Evaporation of water at 80° C.
2. Evaporation of water with oil film at 80° C.
3. Absorption of oxygen from air with alkaline pyrogallol at 25° C.

Figure 3. Typical Curves Obtained with Automatic Recording Balance.



Curve 4. Decomposition of $KClO_3$ with 1% MnO_2 at 368° C.
5. Decomposition of $KClO_4$ at 600° C.

Figure 4. Decomposition Rate Curves.

the metal tube. One can also suspend the reaction vessel in a small electric furnace for work at high temperatures.

Various means of insulating a heating unit from the balance must be employed depending upon the type of apparatus used. The nichrome rod on which the reaction vessel is suspended usually would extend through holes cut in insulating baffles. Baffles of various types are also required when one desires to absorb gases other than those in air.

Figures 3 and 4 are curves plotted by this balance to illustrate some of the types of studies that can be made. Curve 1 (Fig. 3) shows a plot of the evaporation of 90 milligrams of water at 80°C. Curve 2 shows the rate of evaporation of 90 milligrams of water plus 1 milligram of mineral oil at 80°C. Comparing the two curves shows the decrease in rate of evaporation of the water caused by a film of oil on its surface.

Curve 3 (Fig. 3) shows the rate at which 3 ml. of freshly prepared alkaline pyrogallol absorbs oxygen and carbon dioxide from the air at 25° C. This illustrates a study of the rate of absorption of gases from the atmosphere. Of particular interest would be curves of the rates of absorption of moisture from air by various types of drying agents.

It would be possible to employ this instrument to obtain rates of absorption of a chosen gas on the surface of a solid by suspending the reaction tube into a bath of the gas. In this case it would be necessary to devise a scheme whereby a gentle flow of gas over the reactant would exclude air from the bath.

Figure 4 shows curves made by this balance for a study in progress. Curve 4 is a plot of the decomposition of a 1 gram mixture of $KClO_3$ with 1% MnO_2 at 368° C. Curve 5 represents partial decomposition of 1 gram of $KClO_4$ at 600° C. For this data a 15 cm. x 12 mm. reaction tube was suspended in a small electric furnace (Fig. 1-b).

This instrument could also be applied to determine optimum conditions for drying of precipitates to constant weight. One would dry the precipitate in the reaction vessel at a given temperature. A plot of the loss of weight would be produced with a straight line resulting when constant weight is reached. Such plots, made at various temperatures would provide data for determining the optimum time and temperature for drying precipitates to constant weight.

Acknowledgment

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