

# LEEDS AND NORTHRUP LTD. pH MEASURING AND RECORDING INSTRUMENTS AS USED IN THE SUGAR INDUSTRY

By S. DE KOCK

In our modern industrial age most products pass through some process which demands accurate control, whether it be temperature, pressure level, pH or any other process variable. Some people refer to these process environments as "industrial weather." How very fortunate for our industrial weather bureau, because we can predict and control "industrial weather" with ultra precision by the aid of high fidelity instrumentation.

Before we can attempt to control any process variable, either manually or automatically, the first essential is to measure our variable with reliability and precision.

For many years the galvanometer was the best secondary measuring element known for determining electrical variables. Instrument development engineers realised that the refinement of primary measur-

ing in this country in 1923. Although this was a new era in instrumentation the accuracy of measurement was still dependant on the nul balance galvanometer.

After many years of continued research the bottleneck of measurement by utilising a galvanometer was removed when Leeds & Northrup fortunately for us, brought their Model "A" Speedomax into the field in approximately 1938 or 1939. This model "A" Speedomax operated entirely without a galvanometer. It was a pure electronic circuit with a direct electric motor drive for the potentiometric nul balancing. The degree of accuracy of this instrument as well as its reliability was phenomenal. Our principals were then fully occupied in manufacturing instruments for war work, but in spite of this, shortly after the war they produced a still simpler

## GENERAL DESCRIPTION OF CIRCUIT

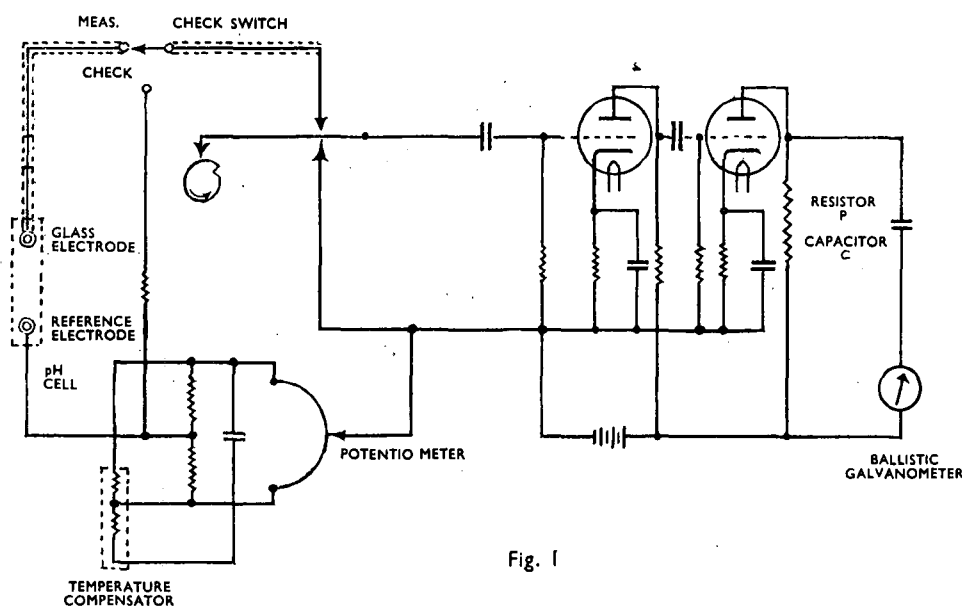


Fig. 1

ing elements such as thermocouples, resistance thermometers, thermopiles, pH glass electrodes, etc., was useless until an instrument more reliable than the galvanometer type millivolt meter was developed.

Leeds & Northrup did their contribution by developing their potentiometer, which was marketed in 1906 and was followed six years later by their potentiometric recorder. We actually have one of these Leeds & Northrup potentiometer recorders in Johannesburg, which was brought into commission

and more modern electronic recorder, which was the famous Model "G."

As we are concentrating on pH measurement today, I will briefly show the development of the Leeds & Northrup Industrial pH Indicator and Recorder.

The first instrument they developed was known as the ballistic recorder. Fig. 1 is a simplified representation of the measuring circuit.

Since the internal resistance of the glass electrode is very great, the cell voltage must be measured without drawing much current from the cell. An electronic circuit is used to transform the cell voltage into galvanometer current. In the electron tube circuit, the current flowing from plate to cathode, or also known as electron flow from cathode to plate (plate current) is a function of the difference in potential between the cathode and the grid.

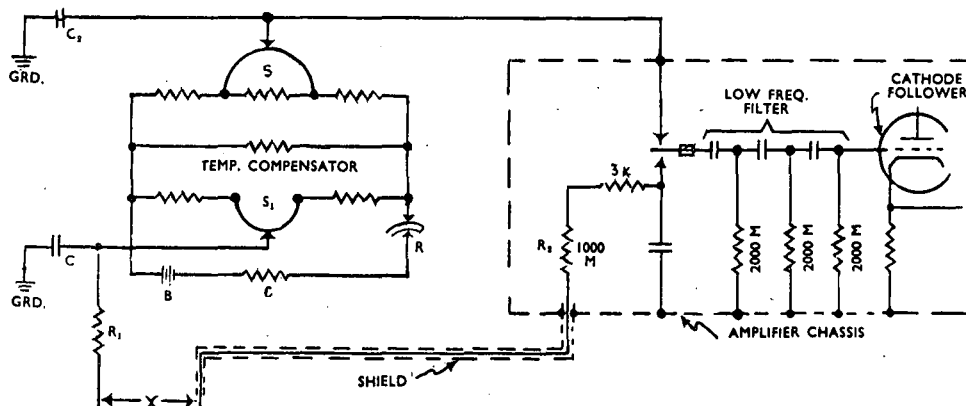


Fig. 2

The plate current through the resistor P will cause a potential drop across the resistor, and current will flow through the galvanometer and capacitor C until the capacitor has charged up to the potential across P. This potential will change if there is any change in the potential between the cathode and the grid of either tube.

The glass electrode recorder circuit is so arranged that the output of the cell is periodically connected in series opposition to the potential of the potentiometer slidewire between the grid and cathode of the first electron tube, through a motor-driven cam switch. If the slidewire potential exactly balances the potential of the electrodes, the grid potential will remain the same regardless of whether the cam switch is in the upper or lower position, but if any unbalance exists between electrodes and slidewire, the grid potential will change as the cam closes one contact and then the other once during every cam-shaft revolution.

If the pH of the test solution changes, the cell output will increase or decrease. The consequent change in grid potential will correspondingly increase or decrease the potential across the resistor P. While the capacitor C is charging or discharging to match the new potential existing across P, a flow of current will cause the galvanometer to deflect. This deflection is picked up mechanically by the micromax mechanism which operates to position the recording mechanism, and to turn the slidewire of the recorder until electrode voltage and potentiometer voltage are exactly balanced.

The Model "A" Speedomax was never used as a pH recorder in our country, but the famous Model "G" Speedomax became a very popular industrial pH indicating and recording instrument. I will briefly give you below the operating principal of this instrument with the basic wiring diagram as illustrated in Fig. 2. This diagram does not show the standardising circuit, but merely the basic measuring circuit.

The battery B supplies current to slidewire S, so that there is a precise known voltage across the slidewire. The slidewire output voltage (i.e. voltage between the slidewire contact and the zero rheostat contact) is connected in series opposition to the unknown voltage X, so that any difference (error) between the two voltages causes a potential to appear across the outside contacts of the vibrator converter. The converter reed V is driven between the outside contacts at line frequency, causing an alternating voltage at very low current, to be applied to the control grid of the cathode follower input. The resultant cathode follower output is amplified through three voltage stages, and a power stage and is finally impressed on the control winding of the balancing motor in the proper phase to drive the slidewire until the voltages are balanced and there is zero signal voltage at the cathode follower grid. The indicating device is mechanically linked to the slidewire and thus indicates the measure voltage. Since this voltage (X) is the output of an electrode assembly or other primary element, the instrument scale may be calibrated in pH or other appropriate terms.

The basic principal of nul measurement is also applied in our latest instrument, the panel mounted indicator.

Incidentally, the Leeds & Northrup Laboratory pH Instrument, operates on the same principal as the panel indicator, which I will describe to you now.

Fig. 3 is a block diagram of the circuit of the panel mounted indicator.

A high gain, low-level, a-c amplifier is the heart of the instrument. Conversion from direct to alternating current by means of a contact type synchronous converter avoids the *conventional d-c amplifier, and its inherent zero drift*. Electronic reconversion to direct current permits the use of overall d-c feedback to stabilise the gain. Sufficient amplification is provided in the a-c amplifier so that the difference between the input voltage and feedback voltage is very small and normal changes in amplification will

This circuitry and the features combined with it provide an instrument which has specific features well worth mentioning.

1. The warm-up time after placing the instrument on the line is short. After about one minute you are in business with this instrument.

2. The sealing of the component parts and the insulation characteristics are such that the device can be operated under conditions of humidity which can well cause failure in other instruments.

3. These indicators are particularly unaffected by

## PART II THEORY OF OPERATION

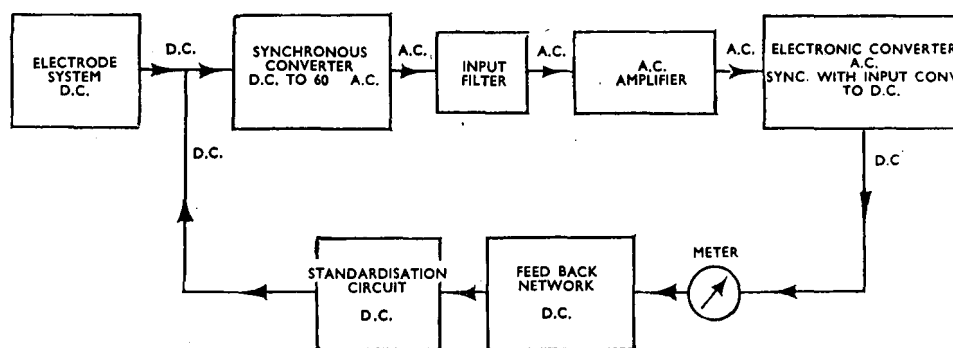


Fig. 3 — Block Diagram of Circuit

produce only small changes in this difference and hence, negligible errors. Since this difference voltage is a small fraction of the electrode voltage, the current flowing in the input circuit is correspondingly small. Hence, the measurement is made under practically open-circuit conditions (null measurement).

The standardisation circuit injects a voltage into the input circuit to adjust the meter pointer position to zero when there is no voltage input from the electrode system. Sufficient adjustment is provided so that the zero position of the pointer can be shifted from one end of the scale to the other. A vernier is provided so that, even with the wide range of adjustment, very fine adjustments are possible. The standardisation circuit also provides the voltage necessary to correct for the asymmetry potential of glass electrode systems.

Fig. 4 is a schematic representation of the input and feedback circuit of the instrument which we have just described. If you compare this with Fig. 2, you will find them very similar.

Some of this circuitry is provided in the form of printed circuits, while the input filter portion is moulded in plastic.

line voltage surges and electrical pick-up and exhibit essentially no zero drift.

4. The accuracy of the meter used in this instrument is relatively high. It is a half per cent. meter. This is, of course, reflected in accuracy of measurement. With less accurate meters accuracy of measurement can only be obtained by the use of an accurate external meter.

5. These instruments are provided for both manual and automatic temperature compensation. The manual compensation is built into the instrument. Automatic temperature compensation is affected by the use of an external connecting compensating thermohm subjected to the temperature of measurement.

6. Special cables are not required to connect the indicator to electrode assembly or recorder. That is to say, the connecting cables necessary can normally be obtained in any radio supply store.

7. These indicators are designed so that they can be used with grounded or ungrounded solutions with no modification in the grounding system.

8. These instruments make use of printed components with conventional wiring and plug-in type electrolytic capacitors. Replacement items are,

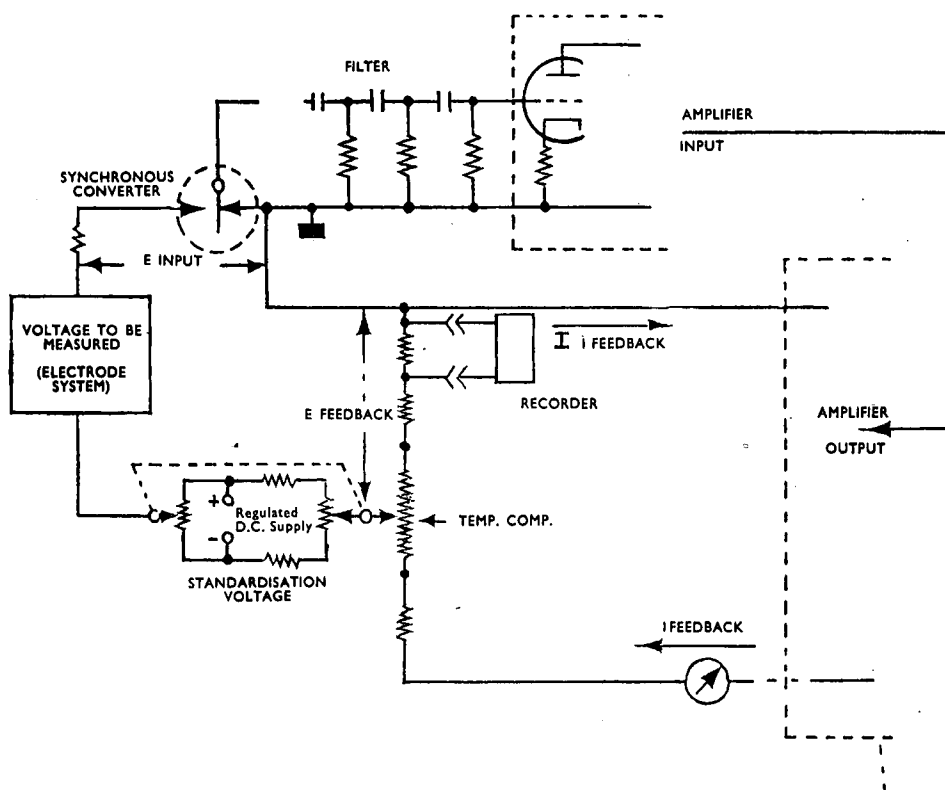


Fig. 4 — Schematic Representation of the Input and Feedback Circuits

therefore, in "unit items," rather than "assembly items."

Both the laboratory model as well as the panel indicator model are designed for and provided with scales for some measurements other than pH. Any potential measurement falling within the range of 1,400 millivolts can be measured with the standard instrument and under conditions of high input impedance (up to 2,000 megohms).

Fig. 5 shows the terminal connections in the panel mounted indicator and indicates some of the versatility of this device. It will be noted that the indicator can be used as a preamplifier to an ordinary millivolt recorder or that a number of remotely mounted auxiliary meters may be operated from the instrument.

Perhaps the best liked feature of these instruments is its high degree of stability and lack of drift. Where measurements are made over a period of time or where the indicator is used directly such as a continuous indicator on continuous process material, it is distinctly advantageous to know that the only standardisation normally required is that of the electrodes. The stability of the meter itself can be depended upon.

Meters of this type have been operating where they are subjected to splashing of water and wash-down of processing equipment. Although the instrument is designed to operate under these conditions,

it is necessary that the rear connections of the instrument be tight and it is suggested that a thin layer of silicon grease be applied to the gaskets behind the terminal box and the door.

The instrument has operated under conditions where water collected on the bottom of the case. Such procedure is not, of course, recommended, but it simply points up the degree of insulation and shielding that has gone into the manufacture of this device.

Under normal conditions, it is advantageous to leave the power supply on the instrument. The switch should be turned off only for long shut-downs or to de-energise the instrument for servicing.

The instruments are supplied with comprehensive directions with a complete listing of component parts with values. This is an aid in making emergency replacements when or where the manufacturer's component parts are not immediately available.

If the operation in the sugar mills is seasonal, it is suggested that in the off-season the equipment be stored in a relatively dry place. It is also suggested that it would be advantageous to have the instrument on the line for a few hours at the beginning of a campaign in order that it might be thoroughly warmed up and dry.

We want to spend a few minutes on discussing glass electrodes and calomel reference electrodes.

We have three specific types of electrodes:

| Electrode        | Marked           | Range °C. |
|------------------|------------------|-----------|
| Std. 1199-22 ... | 2 white dots ... | 50—90     |
| Std. 1199-32 ... | 1 black dot ...  | 10—60     |
| Std. 1199-33 ... | 1 blue dot ...   | 0—20      |

It is wise to use the electrodes for recommended temperatures, the temperature range may be exceeded under some circumstances, but the following should be considered:

1. The electrical resistance of the glass bulb increases logarithmically as the temperature decreases. Very high resistances (2,000 megohms or more) for which the electrical circuits may not be suited may be encountered at temperatures much below the recommended minimum.

2. At temperatures above the recommended maximum, excessive sodium ion corrections may be encountered with alkaline solutions. Also the glass bulbs are more soluble at high temperatures which results in shortened life. When the temperature exceeds 90°C., the calomel may slowly be reduced to metallic mercury with eventual complete failure of the electrode.

Before the electrodes are put into commission it is advisable that they are conditioned by soaking the bulbs in distilled water or a seven-eighths buffer solution for a few hours at approximately room

temperature. This procedure should also be followed with electrodes which have become dry during periods of use.

When an electrode becomes sluggish, it is possible to correct this by immersing the electrode in a 20 per cent. solution of ammonium bifluoride (in a rubber or waxed beaker) for about one minute at about 250°. The amount of glass removed by such a treatment should be small and there is very little change in resistance. Only a very slight surface etching ever results. The electrode, after the bifluoride treatment, should be thoroughly washed and stored in the borax buffer prior to use.

Electrodes not in use can be stored in a vertical position with the bulb immersed in distilled water or pH 7 buffer solution again. Caution should be exercised when cleaning the terminals with alcohol, that some of the alcohol does not drop into the solution in which the bulbs are soaking or immersed for measuring, or even touch the bulbs with the alcohol-soaked cloth. I refer to cleaning the terminals with alcohol.

I would like to stress this point, whenever you are fitting new electrodes or making an adjustment or alteration of any kind, be perfectly sure that no moisture from the hand is deposited on any terminal connection. This is of course rather difficult, and after such work has been completed, take pure

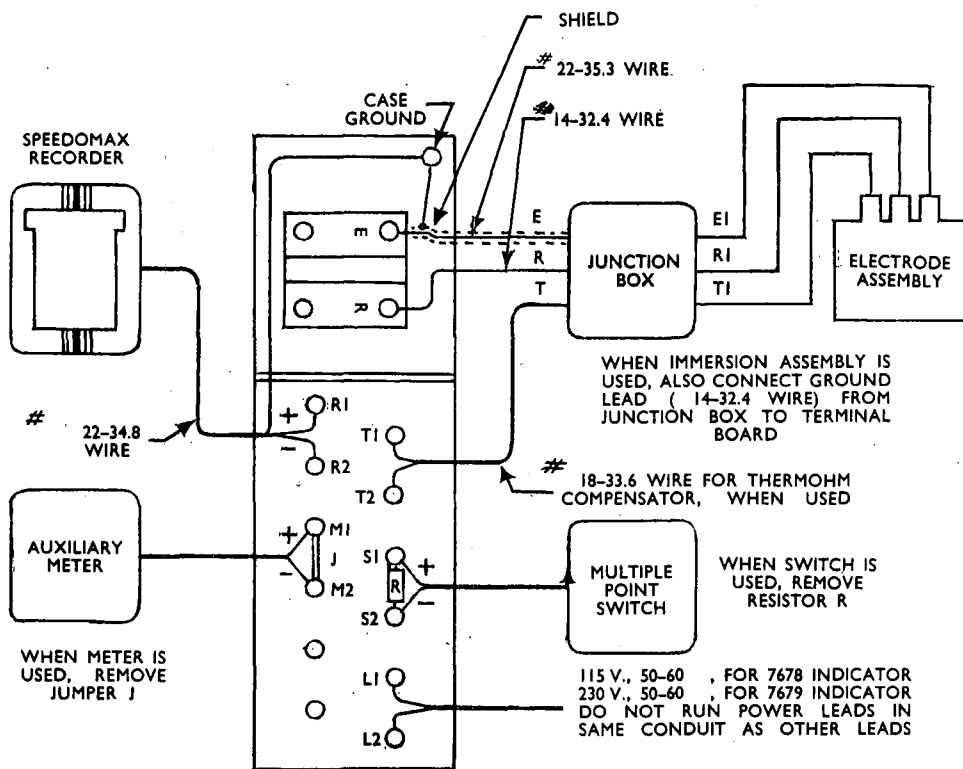


Fig. 5 — Wiring Connections to Indicator Terminal Board

alcohol with a small brush and clean off the terminals to ascertain that no moisture remains behind.

This applies to terminals in junction boxes at the measuring points along the line, or in the instrument. I would like you to take special note of the fact that on numerous occasions difficulties are experienced with pH instruments due to this factor, which appears an insignificant point.

As regards calomel reference in electrodes, the most important point to watch is that sufficient KCl solution is in the reservoir. It is advisable to have undissolved crystals in the reservoir as well. Here again keep the connecting post of the internal element clean and free from moisture from the hands, or any moisture for that matter.

Caution should be exercised that no precipitate forms in the KCl at high temperature. Excess of this precipitate may raise the resistance of the electrode, and give erratic records. It is better to clean out the reservoir immediately and replace with fresh KCl, as KCl crystallises in the liquid junction. After a period of dry storage this condition can be corrected by filling the salt bridge with KCl solution and allowing it to stand overnight in

air. If this method fails, you can actually boil the bridge in water for approximately two hours, which will dissolve the crystallised KCl.

As regards the installation of electrodes, I feel it advisable to use a good type of flow unit, as this flow unit provides a water-tight compartment above the electrodes with the leads leading into a water-tight junction box.

As you know, moisture is our greatest enemy on the electrode connections in pH measurement. From the flow unit to the instrument conduit should be used, preferably overhead and not under floors and platforms, as moisture can easily collect in the loops.

Another important point is that the conduit should be installed with a slight slope, and at both ends compound sealing boxes would be desirable. Although shielded cable can conduits are used, it does not shield the leads from all electrical interferences, it only shields for electro-static waves, but cannot shield against electro-magnetic waves, in other words try to avoid installing electrodes close to switchgear, motors, welding plants and other electric machinery.