A PRESSURE-OPERATED SAFETY CIRCUIT FOR USE IN HIGH-VACUUM SERUM-DRYING PLANTS

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(With 2 Figures in the Text)

The successful operation of the Greaves-Adair (1939) and similar processes for drying blood serum by sublimation from the frozen state depends on the maintenance of an adequate vacuum in the desiccating chamber. If the pressure in the region of the refrigerated condenser coils should rise materially above the operating range of 0·10–0·05 mm. Hg (the vapour tension of ice at −40 to −45° C.), the rate of sublimation from the frozen serum will diminish and the serum will tend to thaw.

A rise in pressure may occur in one of two main ways. In the first place a fault in the mechanical refrigeration cycle may cause a rise in temperature and hence in water-vapour tension at the condenser surface. Secondly, an air leak or a failure of the vacuum pump or its motor may lead to an increase in the pressure of fixed gases in the system.

Fusion, especially of the peripheral layers of the serum, indeed occurs very readily, for the serum is normally receiving from electric heating elements at least enough heat to replace its latent heat of sublimation, a quantity which is very large (670 cal./g.) in comparison with the amount of heat (about 8 cal./g.) required to raise the temperature to fusion point. It follows that if the current to the electric heaters is cut off at the first sign of danger the onset of fusion will be considerably delayed.

Greaves & Adair measure pressure with the well-known Pirani vacuum gauge which utilizes the fact that the thermal conductivity of a gas falls sharply over a pressure range of about 1·0–0·0001 mm. Hg. This is a convenient range, and the Pirani gauge has the additional advantage of being responsive to both fixed and condensible gases.

The object of this paper is to describe a simple form of Pirani gauge which serves three ends, viz. (1) to measure pressure, (2) to give a continuous record of pressure when used in conjunction with a recording galvanometer, and (3) to operate an electromagnetic relay which breaks the electric heating current to the serum if the pressure rises above a critical level.

Kaye (1927) pointed out that the Pirani gauge 'lends itself to self-recording and automatic control devices', and in their original experiments Greaves & Adair used the familiar galvanometer-operated relay in conjunction with thermocouples.

The device therefore embodies no new principles, but it has proved its value in a large serum-drying unit where continuous expert supervision of the plant throughout the 72 hr. drying cycle has so far been impracticable.

CONSTRUCTION OF APPARATUS

(See Fig. 1)

(1) *The gauge.* The gauge filament is that of an ordinary 60 W. (gas-filled) electric light bulb which is connected with the vacuum pumping line. The resistance of the filament is measured at room temperature, and by applying the thermal coefficient of electrical resistance of tungsten (0·0045 per ° C.) its resistance at 110° C. is calculated. A fixed resistance of this value (110 ohms in the present instance) is constructed by winding fine gauge (38 B. & S.) enamelled constantan wire on a fibre rod. This resistance and the filament of the bulb form adjacent arms of a Wheatstone bridge, the remaining arms each consisting of a fixed resistance of 35 ohms. The bridge galvanometer is a micro-ammeter giving full-scale deflexion from a central zero with 60 μA. A more sensitive galvanometer would be an improvement. Current is supplied to the bridge from a 6 V. accumulator through a variable resistance of 0–500 ohms. The potential drop across the bridge is measured with a voltmeter reading from 0 to 10 V., the sensitivity of which has been increased by reducing the series resistance from 10,000 to about 3300 ohms. The bridge can only be balanced, using the variable resistance, when the bulb filament is at 110° C. and the potential required to maintain this temperature varies with the thermal conductivity and hence with the pressure of the residual gas in the bulb. The gauge is calibrated by sealing the bulb to a dry gas-tight vessel connected with a vacuum pump and a McLeod vacuum gauge, and comparing the potential drop across the balanced bridge with the (absolute) readings of the latter at various pressures. The scale of the voltmeter is then reinscribed in mm. Hg.

Finally, the bulb is connected to the vacuum system. A satisfactory union can be made by heating and removing the metal cap, breaking the tip off the central glass nipple, and sealing into the stem a length of copper tube with Apiezon W wax, the leads to the filament being insulated with strips of mica. To take a reading the bridge is balanced by the external variable resistance and the result read on the voltmeter scale. The gauge has an approximate range of 0·8–0·001 mm. Hg, but the response is rather sluggish below about 0·01 mm. The same bridge can be used to measure pressure in any number of desiccators, provided that bulbs of identical pattern and resistance are used.

(2) *Recording pressure.* To obtain a continuous record of pressure it is only necessary to fix the potential applied to the bridge and to use the recording galvano-

meter to record the degree of imbalance of the bridge at various pressures. The original bridge galvanometer may conveniently be retained and transfer from one instrument to the other effected by a two-pole, double-throw switch, the leads to the recording galvanometer being suitably shunted. Our recording galvanometer is a six-point Siemens-Elliott electrically operated instrument giving full-scale deflexion from a central zero with 20 μA. The circuit reserved for recording pressure required additional resistances of 10 and 1000 ohms in parallel and series respectively.

The potential across the bridge is set to correspond to a pressure of 0·045 mm. Hg by adjusting the external variable resistance. Theoretically this should not be done until the pressure in the system has actually fallen to this level. In practice, however, the adjustment may be made as soon as the pressure approaches the working range of the plant.

remaining five galvanometer tracings. If desired the relay system may be adapted to restore the heating current if the vacuum improves.

If two desiccators are in use, the same recording galvanometer circuit and cut-out system can be operated by the two Pirani bulbs, through a rotary switch, synchronized with the selector switch of the galvanometer, which throws each bulb alternately into the bridge circuit. With the Siemens-Elliott meter a simple switch can be made from paired contacts closed by a cam on the axle carrying the circuit indicator, which rotates through 30° between each circuit. This saves duplication of apparatus but has the minor drawback that failure of one plant will cut off the heat supply to both. If a recording galvanometer is not available, a photo-electric cell in conjunction with a shunted mirror galvanometer across the bridge could be used to operate a safety relay.

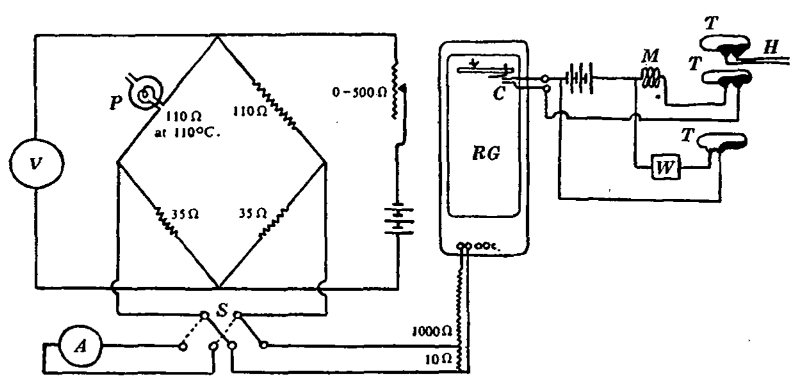

Fig. 1. Diagram of circuit (see text). *P*, electric light bulb; *V*, voltmeter; *A*, micro-ammeter; *S*, two-pole, double-throw switch; *RG*, recording galvanometer; *C*, contacts operated by galvanometer; *M*, solenoid operating mercury tip switches (*T*); *H*, leads to mains a.c. supply and serum heating system; *W*, alarm buzzer.

The tracing recorded in the above way gives a reliable and sensitive indication of pressure in the system. As long as the desiccator is gas-tight and the vacuum pump free from water, the record reflects the slightest change in the temperature of the refrigerator coils, showing a steady improvement from 0·08 to 0·035 mm. Hg towards the end of the drying process when the load on the refrigerator is lifting. These figures are slightly lower than the theoretical values as calculated from the observed temperature range of the condenser, owing to the position of the gauge bulb between the condenser and the vacuum pump. The smallest air leak is immediately apparent on the record.

(3) *Safety relay.* A pair of contacts made from thin annealed silver tape is so mounted in the recording galvanometer that whenever the pressure rises above 0·25 mm. Hg the descending galvanometer needle closes the contacts. This completes a battery circuit operating an electro-magnet which releases a hinged board carrying a mercury tip-switch through which the current passes from the mains to the serum-heating elements. Accessory tip switches serve to break the relay current and to sound an alarm buzzer. It is of course necessary that the region of the chart corresponding to the danger zone of pressure shall not be traversed by any of the

DISCUSSION

Although the Greaves-Adair plant is automatic in operation, the possibility of mechanical breakdown can never be completely excluded and the quantity of serum at stake is very large, amounting in the latest type of desiccators to 40 l. or more. The use of residual pressure to activate a safety relay is logical in that a rise of pressure is the earliest indication of danger from almost any kind of breakdown except overheating of the serum due to a fault in the electrical system.* The main advantage of the relay is that in greatly delaying the onset of fusion it enables an unskilled attendant to summon the operator of the plant who will generally have ample time either to locate and rectify the fault or to return the serum to cold storage.

The extent of this period of grace may be illustrated by an actual incident which occurred at a time when the unit consisted of two desiccators evacuated through a common pipe line by two Cenco 'Megavac' pumps. The first desiccator, containing nearly dry serum and with a moderate quantity of ice on the condenser, developed

* This danger could be covered by a thermocouple operating the same relay through another circuit of the recording galvanometer.

a serious refrigeration failure which rectified itself 6½ hr. later. During this time the condenser temperature (Fig. 2, *C* 1) rose, defrosting occurred, and the oil of the vacuum pumps became heavily contaminated with water. The remaining tracings in Fig. 2 refer to the second desiccator which had been loaded with 11 litres of serum 7 hr. previously. The condenser temperature (*C* 2) remained at −40° C., but the pressure (*P* 2) rose steeply and operated the relay, at that time set at 0·3 mm. Hg, causing a fall in the heating stage temperature (*H* 2) at point *A*. It will be observed that the serum temperature (*S* 2) rose but slowly and after 6 hr. was still below −10° C. Thus the vacuum pumps in spite of contamination with water were able in this instance to maintain a low enough pressure to prevent fusion of the serum.

SUMMARY

A form of Pirani vacuum gauge is described for obtaining a continuous record of the total residual pressure in a high-vacuum serum-drying cylinder of the Greaves-Adair type. The circuit incorporates an automatic relay which breaks the electric current heating the frozen serum if the pressure rises to a dangerous level, and so delays materially the onset of fusion of the serum.

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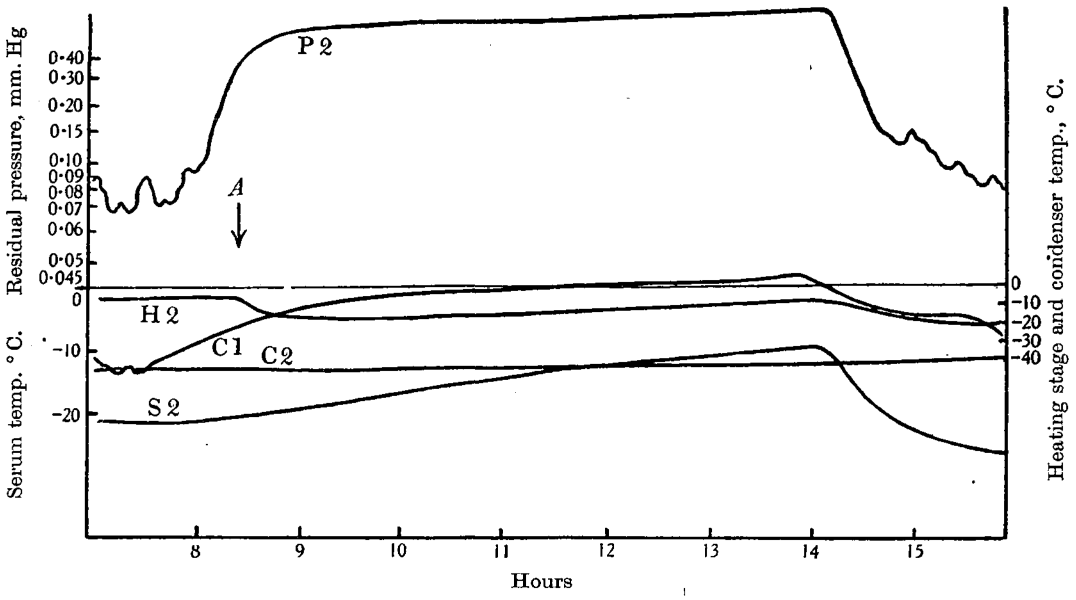

Fig. 2. Effect of a refrigeration failure on the pressure and on the temperature of the serum, heating stage and condenser (see text): a tracing from the chart of the recording galvanometer. *C* 1, condenser temperature in plant 1; *C* 2, condenser temperature in plant 2; *S* 2, serum temperature in plant 2; *H* 2, heating stage temperature in plant 2; *P* 2, total residual pressure in plant 2.

REFERENCES

Greaves, R. I. N. & Adair, M. E. (1939). *J. Hyg., Camb.*, **39**, 413.

Kaye, G. W. C. (1927). *High Vacua*, p. 144. London: Longmans, Green and Co.

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