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An Automated Oxygen Absorption Instrument

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Synopsis

Oxygen absorption is a widely used test for measuring polymer stability and antioxidant effectiveness. Commonly, periodic measurements are made using a mercury buret to measure the rate at which oxygen is taken up by the polymer. We designed and built a safe, dependable apparatus that continuously monitors the reaction of a hydrocarbon polymer with oxygen. The instrument operates by recording the frequency with which small, known volumes of oxygen are introduced into the sample tube to maintain a pre-established pressure in the tube. No operator time is required during the test. The instrument readings are readily converted to standard oxygen absorption curves.

INTRODUCTION

The oxygen absorption test is frequently used to determine the susceptibility of a polymer to oxidation or to measure the effectiveness of antioxidants. There are numerous disucssions of this test and the results in the literature.[1] The instrument described by Shelton and McDonel is widely used for these measurements.[2] However, it suffers from at least two disadvantages. First, each sample requires a mercury buret; our 28-station instrument contained more than 200 pounds of mercury. Second, each buret must be read regularly; this presents a problem overnight and on weekends in an untended laboratory. To eliminate these deficiencies, we developed a simple, reliable, and safe system to automatically record the data from the oxygen absorption instrument.

Other methods have been described for automating oxygen absorption instruments.[3] Generally, these are single station devices involving complex electromechanical systems. The cost of adapting such systems to a 28-station instrument would be prohibitive. The system described here readily attaches to the oxygen absorption instrument described by Shelton and McDonel. No change in sample handling or mounting is required.

RESULTS

We devised a system that automatically measures and records the amount of oxygen that must be admitted to the sample tube to maintain a pre-established pressure. As each small portion of oxygen, approximately half a milliliter, is admitted to the sample tube, an event is recorded for that tube on an event recorder. A plot of cumulative events versus time results in a

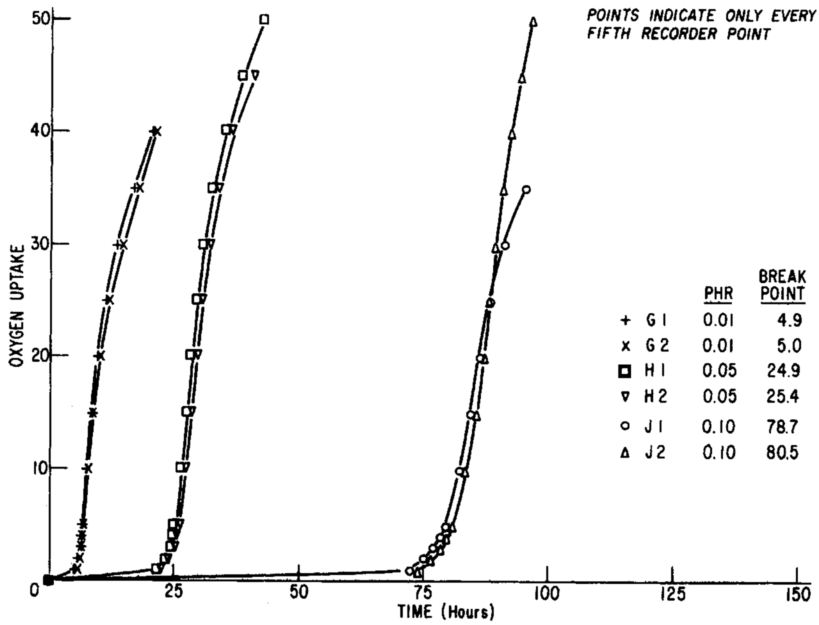


Fig. 1. Oxygen absorption at 140°C of polypropylene containing commercial antioxidants.

typical oxygen absorption curve for a polyolefin (see Fig. 1). On Figure 1 only every fifth recorder point is plotted. Therefore, much more data are available if it is necessary to include more detail in the curves.

The completed oxygen absorption instrument with 28 test stations is shown in Figure 2.

Operation. The polymer sample is placed in a large tube contained in a heated block. The sample geometry is dictated by the physical properties of the polymer. The tube also contains a small amount of 5 Å molecular sieve. The tube is attached to the mercury switch as shown in Figure 3. The three-way stopcock is turned to connect the sample tube to the vacuum-oxygen source. A vacuum is drawn on the sample tube and then oxygen is admitted. This is repeated two more times. By using a manifold, all stations in the instrument are evacuated and charged with oxygen simultaneously. The stopcock on the reservoir bulb is closed and the three-way stopcock is turned to connect the mercury switch to the sample tube. The switch on the controller is turned on and the mercury level adjusted to contact electrode A. As the polymer reacts with oxygen, the mercury level falls on the electrode side of the U-tube. When the mercury falls from electrode B, the solenoid valve opens and oxygen is admitted until the mercury is forced to contact electrode A. This operation is recorded as a spike on the event recorder.

Apparatus. A schematic diagram of the system for a single position is shown in Figure 3. The system consists of: (1) power supply, (2) control box, (3) solenoid valve, (4) mercury switch, and (5) recorder.

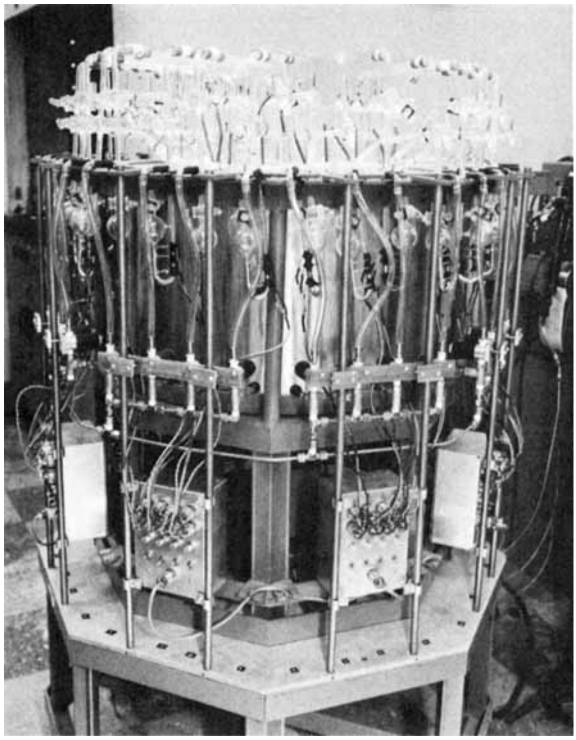


Fig. 2. Automated oxygen absorption instrument.

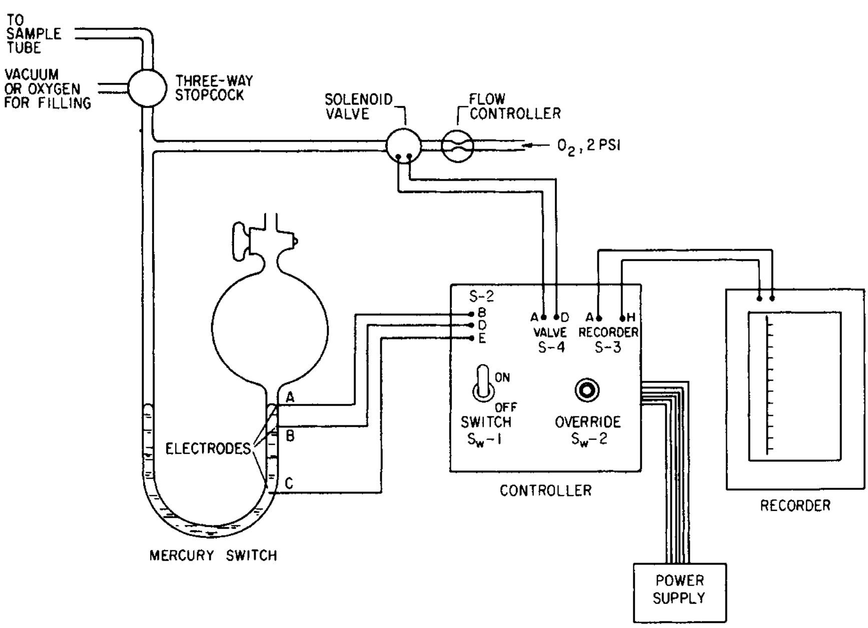
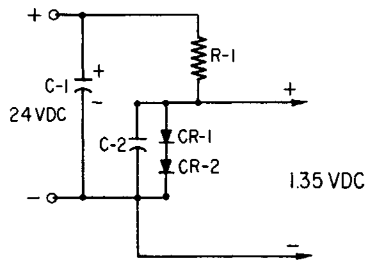


Fig. 3. Schematic-automated oxygen absorption instrument.



PARTS LIST

CR-1,2 = IN-5062 DIODE

C-1 = 40 μfd at 150 VDC CAPACITOR

R-1 = 1 KΩ 1/2 WATT RESISTOR

C-2 = 20 μfd at 50 VDC CAPACITOR

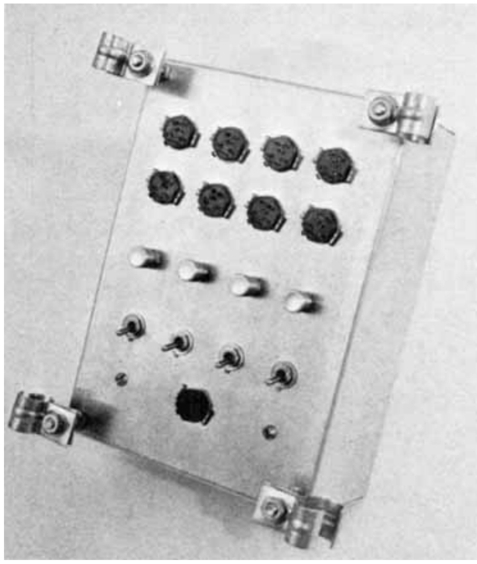
Fig. 4. 1.35 V dc power supply.

Power Supply. The power supply provides 110 V ac, 24 V dc, and 1.35 V dc to each of the seven control boxes. An Acopian Model 24U300 unregulated power supply provides the 24 V dc. The schematic for the 1.35 V dc power supply is shown in Figure 4. The power supply is connected to each control box by a six-conductor cable.

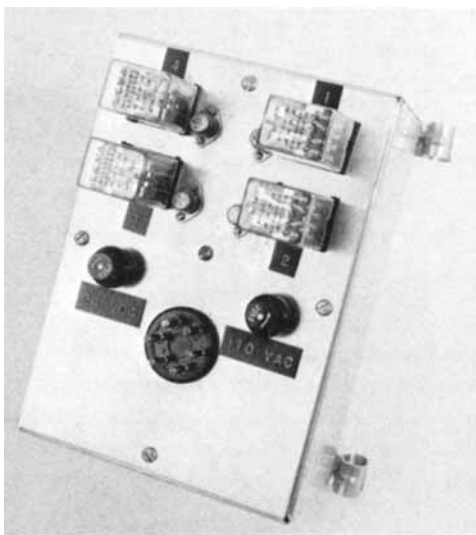
Mercury Switch. The mercury switch (Fig. 3) regulates the introduction of a specific volume of oxygen into the sample tube. It is also designed to permit evacuation and filling operations during the set up procedure. The mercury bulb is sealed with a stopcock. This serves two purposes: A reference pressure is established over the mercury when the stopcock is closed, which is unaffected by changes in atmospheric pressure. The stopcock also isolates the mercury from the atmosphere, thus eliminating a potential health hazard. The lowest electrode (C) is maintained at a constant 1.35 V dc. At this voltage there is no spark when the mercury contacts or separates from the two upper electrodes (A, B). The two upper electrodes determine the volume of oxygen admitted to the sample tube during each cycle. We spaced them for about 0.5 ml of oxygen.

Solenoid Valve. The solenoid valve is a Skinner Model B 110 V ac miniature stainless steel solenoid valve. The valve must be thoroughly cleaned for use with oxygen. The rate of flow of oxygen through the solenoid valve is regulated by a needle valve. The oxygen is supplied at 2 psig from a gas cylinder.

Control Box. The control box (Fig. 5) contains the electronics for coordinating the operations of the other sections. The schematic diagram for the control box is shown in Figure 6. The most important feature of the controller is that the control circuit is a safe, low-voltage dc circuit. This circuit, through a transistor, triggers the relay which operates the recorder and oxygen inlet valve. Each control box controls four sample positions in the oxygen absorption instrument. The front of the control box contains jacks for the mercury switch and solenoid valve for each position. A single jack connects all four positions to the recorder. A momentarily-on push button switch activates the solenoid valve. This is useful when setting up the instrument. Components subject to failure, relays and transistors, plug into jacks on the rear of the control box. Fuses for the 24 V dc and 110 V ac are also located there.



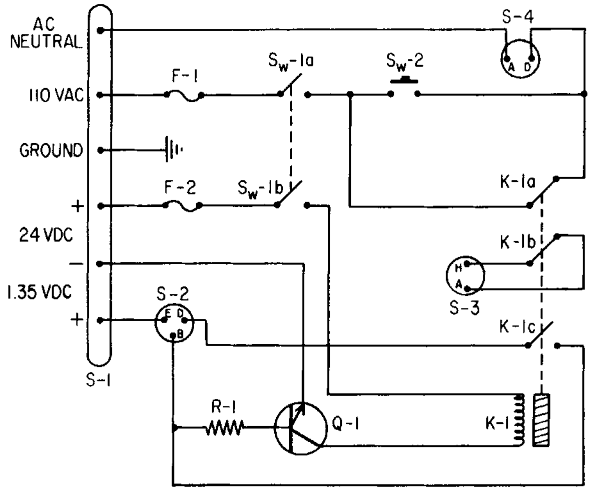
(a)



(b)

Fig. 5. Control box (a) front and (b) rear.

Recorder. The recorder is an Esterline-Angus 20-point event recorder. When the mercury switch operates the oxygen solenoid valve, the recorder pen is activated. We know the volume between the two upper electrodes of the mercury switch. The mercury switches were constructed for 0.5 ml between electrodes. Therefore, the recorder indicates when this known amount of oxygen was admitted to the sample tube.



F-1 = AGC 3
F-2 = AGC 1/2
K-1 = Sigma 67R4-24DC, 4PDT-3 Amp
Q-1 = 2N696 NPN Transistor
R-1 = 910 Ω 1/2W Resistor
S-1 = Amphenol 78-PM6
S-2 = Amphenol #126-198 (Mercury Switch)
S-3 = Amphenol #126-198 (Recorder)
S-4 = Amphenol #126-198 (Valve)
S_w-1 = JMT-223 Toggle Switch DPDT
S_w-2 = PB-126 Pushbutton Switch

Fig. 6. Control box schematic (one position).

CONCLUSIONS

We developed a low-cost multistation instrument that automatically measures the rate of reaction of hydrocarbon polymers with oxygen. This safe, simple apparatus provides reliable, accurate data with a minimum of operator time. The possibility of operator error is greatly reduced. Further, samples are monitored continuously providing "around the clock" readings.

References

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3. M. E. Cain and G. T. Knight, *Chem. & Ind.*, 1125 (1971); H. B. Tinker, J. H. Craddock, and F. E. Paulik, *Rev. Sci. Instrum.*, **39**(4), 590 (1968); J. Scanlan and J. R. Dunn, *J. Sci. Instrum.*, **38**(1), 28 (1961).

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