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MORE HINTS ON SPURIOUS REDUCTION

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An earlier report (S. Sutton and D. Zimmerman, 1977, Ancient TL No.1, p.7) has stressed the importance of reducing TL oven oxygen levels in avoiding significant spurious signals. Additional dangers of oxygen contamination are present when the "oxygen-free" inert gas flows to the oven through lengthy sections of tubing. This note reports experiences with a long nylon gas line, and shows that the use of copper tubing in similar circumstances is highly preferable.

Oxygen contamination was measured at the end of a 40 m length of plasticised nylon tubing (Type II, supplied by Enots Ltd, Lichfield, Staffs, England) of inner diameter 5 mm and 1.2 mm wall thickness, which was fed from a high purity Argon gas bottle.

The oxygen measuring device was a Couloximeter, (obtainable from Chemical Sensor Development, 910 Franklin Terrace, Minneapolis, Minnesota 55406) described by P.A. Hersch, (1973), August 1973, American Laboratory, p.29. Upstream of the meter the line incorporated a by-passable column of oxygen absorber, a humidifier, and a micro-electroyser capable of producing oxygen for calibration purposes. During measurements the gas flow through the meter was held at a fixed value of approximately 40 cc/min. Back diffusion of oxygen was prevented by a non-return valve at the vent. The line could be additionally vented immediately upstream of the couloximeter.

Equilibrium oxygen contamination levels in the argon gas were measured as the flow rate through the nylon line was varied between 400 cc/min and 4 ℓ/min. Fig. (1) shows two plots of contamination levels Vs. the reciprocal of the flow rate, one for a line excess pressure of 11 lbs/in^2, the other for 23 lbs/in^2. Neither of the plots shows significant departure from linearity, implying that the rate of ingress of oxygen into the gas line is independent of gas flow rate. Oxygen ingress rates were 1.7 $\mu\ell$/min at STP per m of tubing at 23 lb/in^2 excess pressure, and 3.2 $\mu\ell$/min at 11 lbs/in^2. (An oxygen diffusion rate of 4 $\mu\ell$/min per m of 'Tygon' tubing is quoted in literature supplied with the couloximeter). A 10 m length of the nylon line was pressure tested at 90 lbs/in^2. No measurable loss of pressure occurred over 24 hours, but, with the line submerged in water, small bubbles formed uniformly over its surface. We therefore believe the observed oxygen ingress to be due to diffusion rather than leakage.

For comparison, similar couloximetric tests were carried out on an all-copper gas line 17 m long of inner diameter 4.9 mm and 700 μm wall thickness. The line was fed by argon gas from the same bottle as used in the previous experiments. At a flow rate of 40 cc/min, an equilibrium contamination of 3 ppm O_2 was measured. This level dropped to 0.8 ppm when the argon flow rate was increased to 1.5 ℓ/min.

A length of nylon coiled tubing has been useful as a flexible connecting section between the gas line and the bottle. This has an extended length of 8 m, and was shown to be responsible for a significant fraction of the oxygen contamination. A nitrogen gas bottle, having a nominal oxygen contamination of 5 ppm, was connected to the all copper line described above. During fast flushing an 8 ppm oxygen level was measured, and at a flow rate of 40 cc/min, 10 ppm. Inserting the coil of nylon tubing in the gas line increased the contamination at the lower flow rate to 20 ppm.

During normal usage, the TL oven is evacuated and then filled with inert gas and flushed at a rate which is generally several litres/min. Periods of usage alternate with longer intervals of no gas flow, the lengths of the intervals depending on the work in hand. It is therefore difficult to estimate the effective flow rate, and the resulting contamination of the gas used in past work. However, these results suggest that our earlier finding that the high flow rate of 5ℓ/min is beneficial in reducing spurious TL levels may result from the ingress of oxygen into the supply line rather than evolution of O_2 from the heated sample, as had been supposed.

Figure 1