SUBJECT PURIFICATION OF RARE GASES IN ARCING BOTTLE

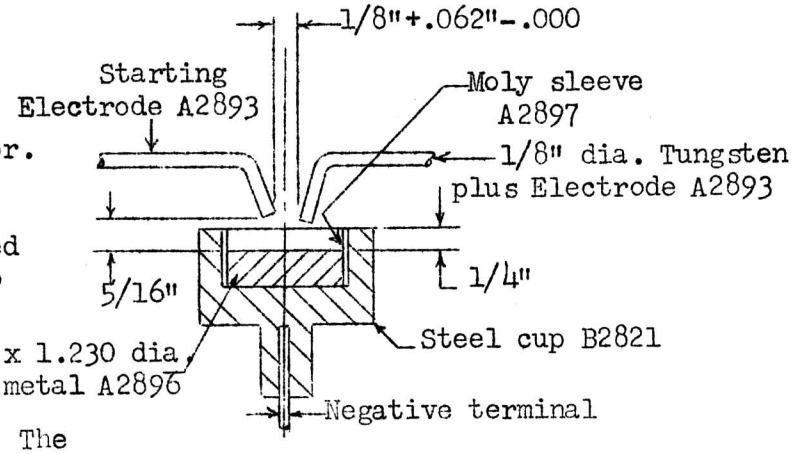
Supersedes former 15-1-2 p. 40-1

When rare gas is purchased in a tank, it is necessary to purify the gas before it is to be used for the filling of tubes. This purification is accomplished by the use of 33-M-47 misch metal and an arc discharge from it. Impurities in the gas, such as oxygen, nitrogen, hydrogen, etc., are removed during arcing by combining with the rare earth metal constituents of misch metal to form stable compounds.

1. EQUIPMENT

a. Glass arcing bottle - Model 702HJ. The arcing bottle consists of a flask of 3 liters capacity which is constructed as shown in Equipment Section drawing No. D2890. The relative position of the electrodes is very important as shown in accompanying sketch. The bottle should have a rough vacuum applied as soon as glass blowing is completed in order to remove water vapor and oxygen. When not in use, all bottles should be sealed off under vacuum in order to prevent deterioration of misch metal and to insure subsequent user that bottle has no leaks.

b. Arcing Bottle Support - The arcing bottle must be supported on a tripod ring stand fastened to floor.



c. Stop-Cocks. The bottle must be connected to glass system through two stop-cocks (part No.B2892, Model 702HJ), which are connected in series with about 3 inches of glass between. The stop-cock barrel must be reground with the finest abrasive or rouge, either by the manufacturer or the glass blower. These stop-cocks must be placed far enough away from arcing bottle, or any other source of heat, so that stop-cock grease does not become overheated. (Do not mount stop-cocks directly over arcing bottle unless a means of artificially cooling the stop-cocks is installed).

d. Rheostat - 100 ohms 500 watts.

e. Ammeter - 0-10 amp. (The ammeter may be taken out of circuit after a rheostat setting for proper current has been determined.)

f. Inductance - 120 turns of #12 cotton covered copper wire wound on 1-1/4" dia. fiber cylinder.

g. Electrical supply consisting of 3000 V. D.C. with ammeter, rheostat and inductance in series. The negative side of supply is connected to misch metal and positive side to electrode closest to misch metal.

*General revision.

1. EQUIPMENT (Cont'd)

h. 150 or 300V. D.C. supply with ammeter and high current carrying capacity rheostat for the circuit. To be used for breaking down crust of misch metal.

i. Spark coil for starting arc. The inductance should be on grounded side of line. It facilitates starting the arc in helium mixtures.

j. Exhaust equipment & McLeod gauge.

2. PROCEDURE

a. After the arcing bottle is connected to vacuum system, grease the stop-cocks as specified in 33-G-17 with Lubriseal.

b. Evacuate bottle to pressure of 1 micron or less.

c. Heat bottle with soft flame. (Protect stop-cocks from heat by placing a wet rag around them).

d. After bottle has cooled, the pressure on McLeod gauge should be less than .5 microns. No glow should be apparent in bottle when spark coil is applied to electrodes of bottle.

e. Close stop-cocks to arcing bottle.

f. Connect tank of gas to vacuum system by means of a rubber tube which is pinched off by a pinch clamp and open the reduction valve to give a pressure of 5-10 pounds.

g. Pump out system and flush gas thru 3 or 4 times by opening pinch clamp to give several centimeters pressure in system. Continue pumping all the time.

h. Apply liquid air to trap.

i. Close stop-cocks to pumps and admit 10-20 cm gas pressure to system. Open stop-cocks to arcing bottle and admit about 20 cm gas to bottle.

j. With full resistance of rheostat in circuit, apply voltage and start arcing by momentarily contacting lead of starting electrode with spark coil. After the arc is operating, admit more gas to the bottle to a total pressure of about 3/4 atmosphere for argon.

k. Close stop-cocks to arcing bottle - then close valve on tank and remove rubber feeder line from manifold.

l. Whenever the gas is supplied from a storage reservoir attached to system, any desired pressure may be admitted to arcing bottle by opening stop-cocks to arcing bottle when a good vacuum exists in the system and stop-cocks to pumps are closed.

m. Regulate the resistance in the circuit to give a current of 5-6 amperes for continuous purification of argon but to value of 2-3 amp. in steady state for helium or helium mixtures (when too high current is used for helium, the cathode will become red hot and the misch metal will boil over and break the glass.)

CHT-2

2. PROCEDURE (Cont'd)

n. Purify the gas by arcing 5 to 6 hours before use. Operate the arc several hours each day during subsequent use. However, the initial purification may be greatly speeded up by passing a large current thru the arc for a short time as described in next item.

o. Each time fresh gas is added to bottle, break down the crust of misch metal by passing 25-30 amperes thru the arc. In general, 10 to 15 minutes is sufficient time to cause melting of misch metal or to remove most of crust. This breakdown of the crust is very easy in helium or helium mixtures because the arc drop is high so that the misch metal can easily be melted. Discontinue the high current as soon as misch metal becomes molten when a fresh surface of misch metal is insured. The current must be kept below that which will melt the tungsten. With argon, the breakdown is not so easily accomplished but high current treatment is absolutely necessary or the arc becomes completely ineffective after a crust has been built up around point of arc source in misch metal.

p. After long useage, the arcing bottle becomes very dark from vaporized misch metal so that it is necessary to replace the misch metal cup from time to time. The frequency of replacement will depend upon the amount of impurities which have been cleaned up in the gas.

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