



The process of reclaiming mercury which may contain foreign solid matter oil, dirt or amalgamated metals such as lead from solder, is herein standardized. Mercury shall be sent to the Chemical & Physical Laboratory to be reclaimed.

1. EQUIPMENT

- a. Cotton cloth 1-1/2 feet square.
- b. Filter paper - 8" to 12" diameter.
- c. Funnel - 4" to 6".
- d. Funnel holder.
- e. One or two liter beakers.
- f. Two liter acid bottles.
- g. Glass tubing about 3/8" diameter.
- h. Rubber " " 3/8" "
- i. Sink with hot tap water.
- j. Compressed air line.
- k. Mercury distillation apparatus with capacity of 110cc/hr. (See p. 2 & 3) consisting of:

- 1. One nichrome heater coil (28 mil nichrome wire - 3.75 amp., 110V. or 415 watts.)
- 2. Alundum cement for insulating heater coil.
- 3. Two 4" D., round, pyrex bulbs.
- 4. One water jacketed condenser.
- 5. About 10 feet of 1/4" pyrex capillary tubing of 1mm to 2mm bore.
- 6. 3/8", 1/2" and 3/4" pyrex glass tubing for connections.
- 7. Two tungsten electrodes, 20-40 mil diameter and about 4" long.
- 8. One pyrex glass stop cock.
- 9. Rubber tubing for vacuum connection.
- 10. One 6" slide wire rheostat, 5 amp., 15 ohm.
- 11. One red, 10 watt, 110 volt lamp.
- 12. One G.E. relay type H.G.-7, 110V., 0.3 a.
- 13. One Bunnell Relay, 100 ohm.
- 14. Dry cells, switches and fuses, clamps and panel boards.
- 15. City water supply and drain.
- 16. Vacuum system.
- 17. 110 volt A.C. supply line.

2. DESCRIPTION OF STILL

A. Mercury Reservoir: A mercury reservoir, Fig. 4, page 3, for holding mercury to be distilled is made large enough to hold a good supply of mercury so that one filling will last several hours of operation. The capillary tubing from boiler to reservoir should extend into a well so that mercury in reservoir may be allowed to almost empty without danger of breaking vacuum system. Mercury is poured into bulb thru a separate opening, conveniently placed on upper side of bulb and loosely stoppered to exclude dust and dirt. Capillary tube extends into reservoir thru a glass tube, top of which should be closed after assembly to exclude dirt.

***Indicates removal of a reference to an obsolete notice.

35/EG



SCHEMATIC LAYOUT OF CONTINUOUS MERCURY STILL WITH AUTOMATIC SHUT-OFF

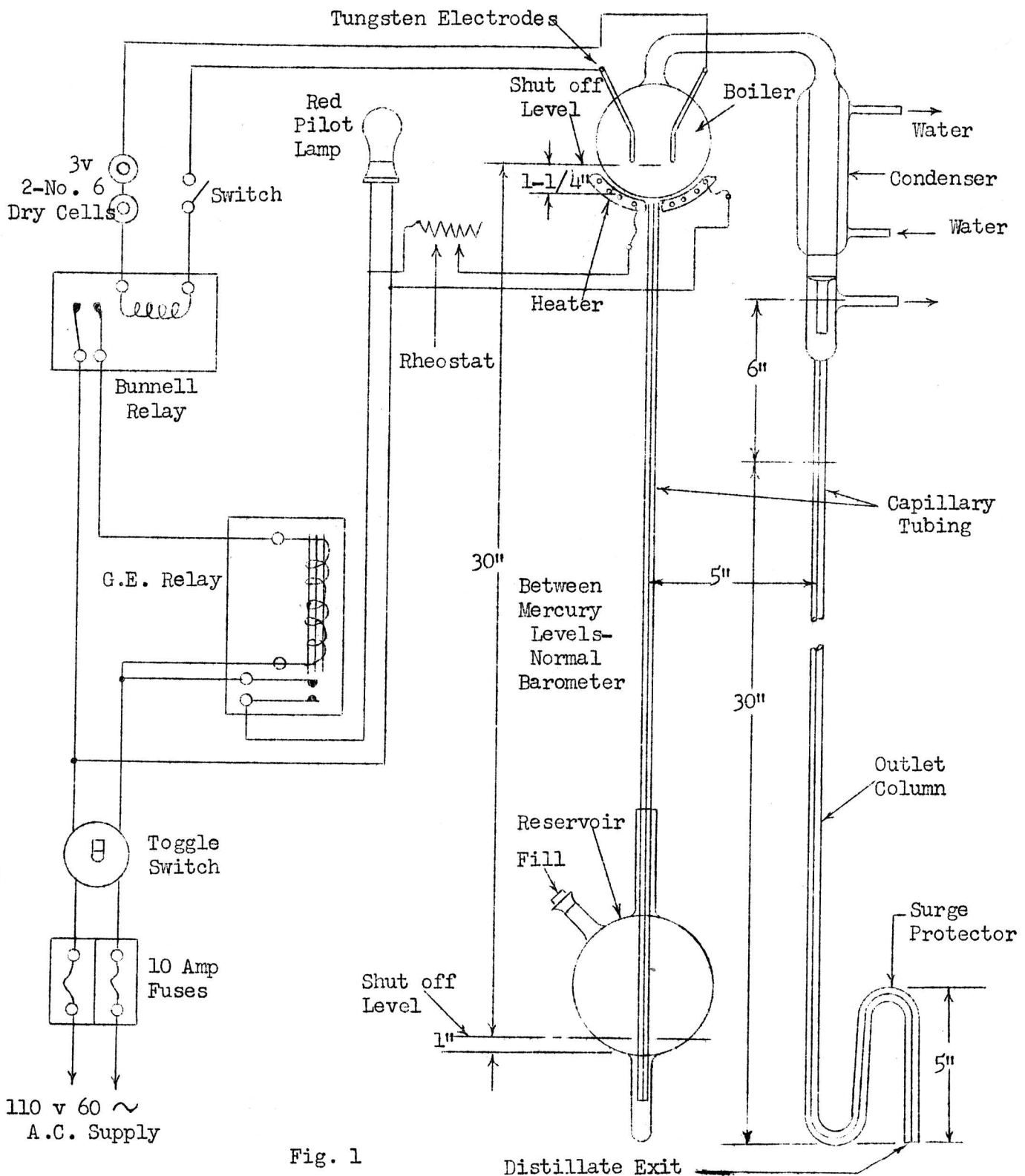


Fig. 1

Distillate Exit

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★ CHANGE
★★ ADDITION
★★ DELETION

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SUBJECT RECLAIMING MERCURY

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DETAILS OF MERCURY STILL

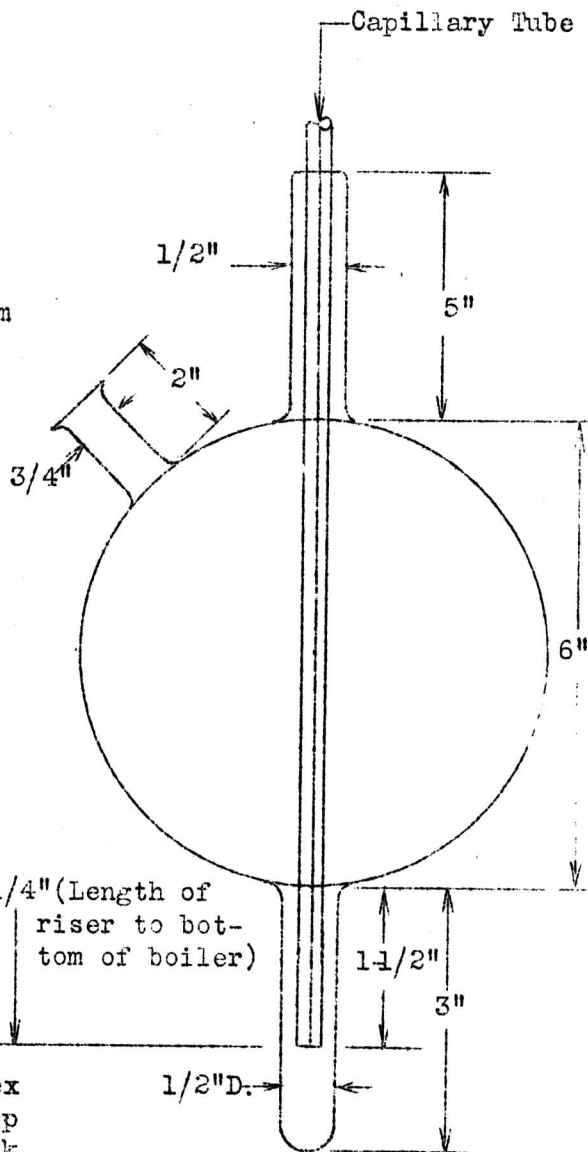
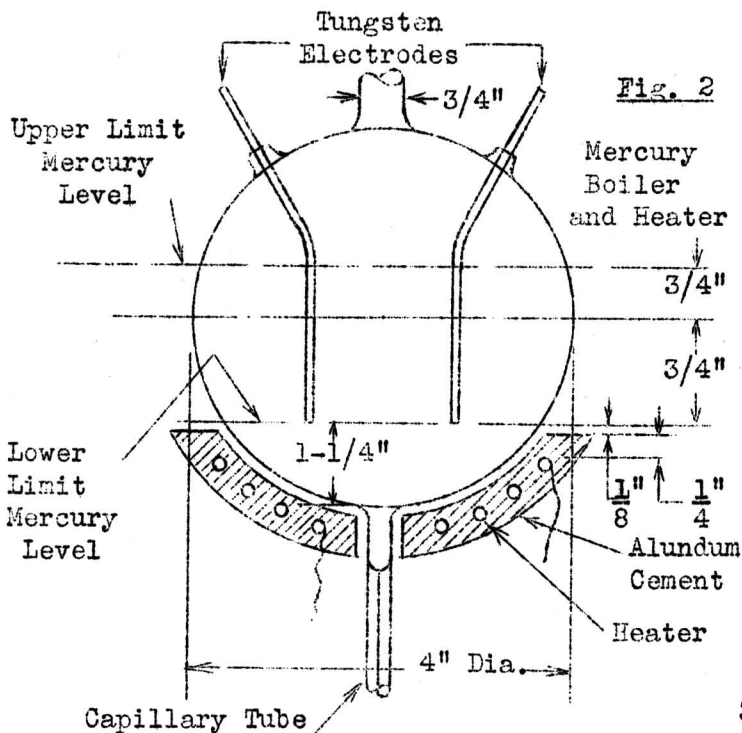
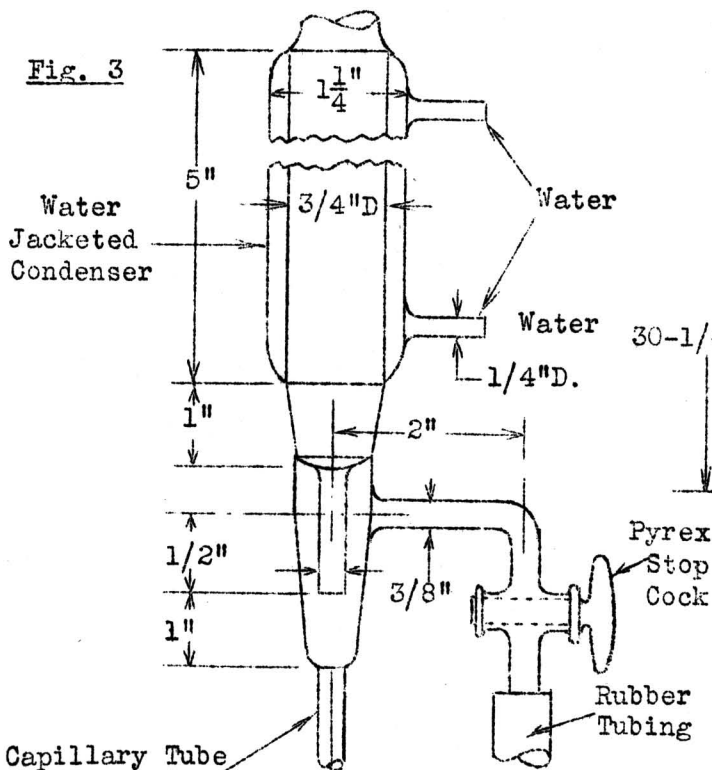


Fig. 4 92S-1177

MERCURY RESERVOIR



Note: Pyrex Glass Thruout



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2. DESCRIPTION OF STILL (Cont'd)

B. Boiler: A boiler, Fig. 2, page 3, contains two electrodes which are a part of electrical circuit to automatically shut off the heating current when mercury level in boiler falls below lower ends of these electrodes. This prevents overheating of exposed glass bulb by heater coil. The electrodes carry a current of only about 30m.a. so their size, 20-40 mil, is selected only for ease in handling.

C. Heater for Boiler: A heater, page 3, is made of 28 mil diameter nichrome wire wound into a 3/16" coil and then imbedded spirally in alundum cement, shaped to fit bottom of boiler. Heater wire must not touch glass at any point. Heater has a 1/2" hole located in center to allow 1/4" capillary tubing to extend thru down to reservoir.

Heater requires about 400 to 425 watts to vaporize approx. 110 cc mercury per hour. At this heat input vaporization of mercury proceeds quietly without bumping or splashing so that only vaporized mercury reaches condenser. Heater, boiler, and tube connecting boiler to condenser should be insulated with asbestos cloth or tape in order to obtain a high heat efficiency.

D. Condenser: A condenser, Fig. 3, page 3, is the ordinary water jacketed type, The lower part, however, is constructed with a separate chamber having a side outlet connection to vacuum pump thru rubber tubing. Side outlet is provided with a stop cock so that vacuum line may be turned on when necessary during operation.

In order to prevent condensed mercury from entering vacuum line opening, the mercury passes thru a nozzle in the chamber which extends one inch below side outlet.

E. Mercury Outlet: Purified mercury outlet column Fig. 1, page 2, is made of capillary tubing and must be slightly longer than barometric column. The lower end is provided with a surge protector so that with any large movement of mercury the column of mercury will at all times be sufficient to maintain barometric height.

Capillary tubing is used so that gases from still which are trapped in the capillary tubing between droplets of condensed mercury are carried along with mercury, the action being the same as in the Sprengle pump and thus the still, once started, will maintain its own vacuum.

F. Automatic Shut-Off: The automatic shut off operates when level of mercury in boiler breaks contact with tungsten electrodes. These electrodes are connected in series with two dry cells and a Bunnell relay. The relay is so adjusted that at the first break in boiler circuit, the relay contact opens and remains open. This is done by adjusting contact gap and tension on armature spring until the magnets have only sufficient force to hold the armature in the closed position but are unable to automatically close the relay if boiler circuit is again completed. This requires, of course, that at the start of the distillation the armature will have to be put into the closed position by hand. The two dry cells need little attention since only 30 milliamperes is required to operate relay.

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Process Specifications

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2. DESCRIPTION OF STILL (Cont'd)

F. Automatic Shut-Off (Cont'd)

In series with the Bunnell relay is a type #G-7, General Electric relay operating on 110 volts A.C. and 0.3 amperes. The heavy current to the heater in the boiler is controlled by this relay and is also varied by means of a 15 ohm, 5 amperes, slide wire rheostat.

A red indicator lamp connected in parallel with heater circuit is lighted when still is in operation.

3. MATERIALS

- A18 Acid Nitric Conc. (Technical)
- A24 Acid Sulfuric Conc. (Technical)
- W7E Distilled Water
- Sodium Dichromate Crystals (Technical)

For preparation of chromic acid solution refer to 34-17-4A.

DANGER

- **MERCURY SAFETY PRECAUTIONS: See S.N. 33-2-10A
- CHROMIC ACID PRECAUTIONS: See S.N. 33-2-7B
- NITRIC ACID PRECAUTIONS: See S.N. 33-2-7C
- SULFURIC ACID PRECAUTIONS: See S.N. 33-2-7C

4. PROCEDURE

- A. Preliminary Cleaning: Strain mercury as received thru a cotton cloth to remove most of loose foreign material. Fold a cotton cloth about 1-1/2 feet square over a beaker, of one or two liter capacity, and pour about 250 cc. of mercury into cloth. Fold ends of cloth together to make a bag and press by hand to squeeze mercury out of cloth and into beaker.
- B. Washing: Place beaker of mercury in sink and wash for about one or two minutes with hot running water, at a rate of flow insufficient to carry mercury globules out of beaker. This removes most of remaining dirt, scum and oil. Water is removed by pouring off as much as possible and soaking up remainder with a clean dry cloth.
- C. Filtering: Fold an 8" to 12" filter paper to fit funnel and punch 3 to 4 holes around end with a pin. Filter mercury into a beaker making sure that no scum or foreign matter filters thru, by discarding last cc remaining on paper. Heavy scums forming from apparently clean mercury generally indicate amalgams are present, in which case prolonged acid treatment will be required.
- D. Acid Treatment: This operation should be done under a well ventilated draft hood. Pour washed and filtered mercury into a two liter acid bottle until about a quarter full. Add diluted nitric acid (1 part concentrated nitric acid to 5 parts of water) until bottle is half to two-thirds full. Bubble air, which has been freed from oil droplets by passing thru a glass or metal tube filled with loose cotton waste, thru the mercury. This is accomplished by inserting a glass tube connected to air hose into the mercury. Allow air to flow for about twenty-four hours at a sufficient rate of speed to moderately agitate mercury.

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4. PROCEDURE (Cont'd)
D. Acid Treatment (Cont'd)

If amalgams are present in quantity, as is indicated by a heavy scum forming from apparently clean mercury, continue air bubbling treatment for at least forty-eight hours.

Finally remove all acid by flushing with tap water. Pour the wet mercury into a beaker, pour out excess water, and wipe mercury dry with clean filter paper.

E. Operation of Still: After assembly and before mounting into position, thoroughly clean still by washing with chromic acid cleaning solution and then rinsing with tap water and finally with distilled water. Dry still before using by drawing air thru apparatus and reservoir by connecting to vacuum line.

Mount apparatus on a metal stand with appropriate clamps and adjust reservoir to proper height.

Half fill reservoir with mercury which has been cleaned as described above, plug outlet end of delivery tube with a piece of clean rubber tubing and glass rod and open stop cock to vacuum. Then keep adding mercury until reservoir is about half full. Turn on condensing water.

Close the battery and 110 volt A.C. supply line switches and move Bunnell relay armature to closed position by hand.

When sufficient mercury has condensed in delivery tube to completely fill column, remove bottom plug, close stop cock to vacuum and the still will continue to operate until it automatically shuts off, provided mercury is allowed to drop to shut off level. To again operate still after it has been shut off automatically, it is only necessary to add more mercury and close relay armature.

All switches may be opened when still is not to be used for some time.

Deposits and scum, which may collect in still, may be removed by cleaning with nitric acid and chromic acid cleaning solutions. Wash the parts with diluted nitric acid. (1 part concentrated nitric acid to 5 parts of water) and then with chromic acid cleaning solution, followed by tap and distilled water rinsing and vacuum drying.

5. RECLAIMED MERCURY SHIPPING CONTAINER

Store reclaimed mercury after distilling in special bottles of glass or stoneware of approx. 400 cc. capacity. Containers should be thoroughly cleaned by following process.

Wash out containers with tap water to remove loose dirt. Remove any broken off corks. Add diluted nitric acid to remove films of mercury and scum, and rinse in tap water. Half fill container with chromic acid cleaning solution and rotate while tilting so that whole surface comes in contact with cleaning solution. Continue for about a minute and pour cleaning solution back into stock container. Rinse with tap water several times and finally with distilled water. Invert bottle, drain and dry in oven at 80-120°C. Stopper bottles with clean new corks.