

# OPERATING INSTRUCTIONS FOR 7470 KETENE-BUTADIENE GENERATOR

Ketene is prepared by the pyrolysis of Acetone and 1,3 Butadiene by pyrolysis of Cyclohexane. The device is essentially that described by Williams & Hurd<sup>1</sup> and Fieser and Fieser<sup>2</sup>.

Chromel A wire is used as the heating element as described in (1) and the element support is kept taut by means of a 20 gram weight suspended at the lower ends as described in (2).

The unit is capable of producing 0.45 mol/hr (19 grms) of Ketene; 350 cc of Acetone will produce approx. 10 moles.

## OPERATION:

Hook the 20 gram weight to the glass loop at the bottom of the element and check the sleeve to see that it moves freely. Insert the element into the previously assembled apparatus and attach the cord to the leads at one end and plug the other end into a source of variable voltage such as a Powerstat®, Variac®, etc. DO NOT TURN POWER ON YET.

Attach a separatory funnel, etc. to the hose connection on the boiling flask and add the Acetone or cyclohexane until the flask is about half full, then shut off the reservoir. Connect the outlet of the trap from which ketene will emerge to what ever other apparatus is being used, and close the trap stopcock.

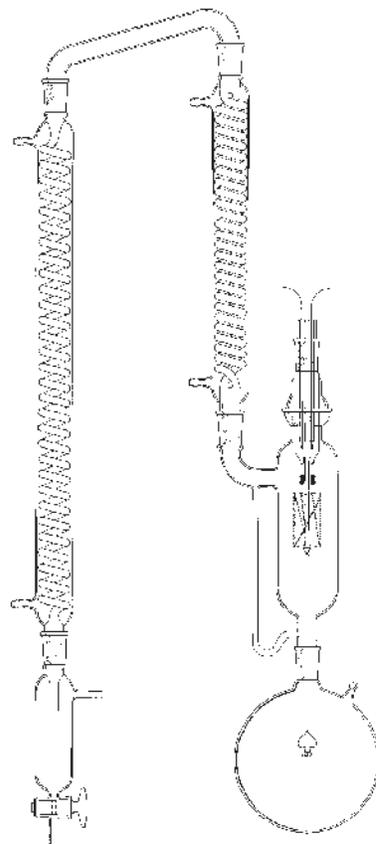
Commence heating and allow the Acetone to reflux from the condenser for five minutes or more to drive air out of the element chamber. Current may now be turned on to heat the element to a dull red glow (700-750°C).

During operation, liquid accumulating in the trap must be removed occasionally; if too much condenses, reduce rate of boiling and/or place an auxiliary trap in an ice bath, before the final trap.

At the end of the run, perform the following operations rapidly in sequence: (1) remove heat from the flask. (2) shut off current to the element. (3) open the trap stopcock.

## CALIBRATION:

The amount of Ketene produced per hour may be determined either by weighing the Acetanilide produced by passing the effluent gas stream through excess Aniline for a measured period of time, or by passing the gas stream through standard alkali with subsequent titrations of the unused alkali. By the second method, the apparatus described was found to deliver 0.45 mole of Ketene per hour. In a continuous run of ten hours, 4.53 moles of Ketene was produced with a net consumption of but 350 ml of liquid from flask A. If the residual liquid and condensate were pure Acetone this would represent a 95% yield, but the figure is too high, for although the liquid is chiefly Acetone, it also contains small amounts of Acetic Anhydride, Acetic Acid and Acetylketene. The lamp should be operated for fifteen minutes to expel air from the system before starting to calibrate the apparatus.



**ACE GLASS INCORPORATED**

**P. O. Box 688 VINELAND, NJ 08360-0688**