# Laboratory Pressure Reaction Apparatus


## ABSTRACT

A laboratory reactor which can be readily built at low cost is described. It provides convenient control of pressure, temperature, and agitation and permits removal of samples under pressure. The reactor has been used to study hydrogenation of 0.5-1.0 kg samples of edible oils at pressures up to 53 psi and temperatures up to 121 C. This equipment should be useful in studying a variety of other reactions of interest to oil chemists.


## INTRODUCTION

In the course of our recent investigation of factors affecting hydrogenation of bovine milk fat (1), the need arose for a suitable laboratory hydrogenation apparatus. We required a reactor that would permit sampling during a hydrogenation experiment and provide convenient control of pressure, temperature, and agitation. Funds were not available to purchase a commercial unit, so we designed a reactor that was built in the departmental machine shop from surplus and readily available components.

## MATERIALS

All parts of the reactor contacting fat or hydrogen are made of stainless steel. The body of the reactor (Fig. 1) consists of a 20 cm section of 12.7 cm (outside diameter) pipe, 0.63 cm wall thickness, type 316 stainless steel. The bottom is a plate, 1.27 cm thick, welded in place. A square flange, 1.27 cm thick, also is welded to the top of the pipe. The cover is a square plate, 1.27 cm thick, that matches the flange and is held in place on top of the reactor by a 1.27 cm bolt at each corner. The motor and stirrer assemblies are supported by a verticle plate attached to a rod welded to the cover.

Constant agitation is provided by a 5 cm split disc impellor with a slight pitch, rotated by an explosion proof electric motor (1/3 horsepower, 115 volt, 60 cycles, 1725 rpm). The motor is connected to the stirrer shaft by a star flexible coupling (clutch). The shaft seal is a type 9T ceramic seal (John Crane Packing, Morton Grove, Ill.). A silicone rubber O-ring 2-349 (Porter Seal, Hayward, Calif.), fitted into a groove machined in the cover, seals the top assembly to the body of the reactor.

The reactor is heated by a 300 watt blanket type heater (Watlow Electric, San Francisco, Calif.) bonded with contact cement to the outside of the chamber and insulated with asbestos tape bonded to the heater. Temperature is controlled with a 18002-21 Fenwal thermoregulator, range -70-315 C. Electrical switches, fuses, and indicator lights are mounted in the control box.

## PROCEDURES

The sample (0.5-1.0 kg) plus catalyst is heated to the desired hydrogenation temperature in the reactor. Cylinder hydrogen is passed through a two stage Matheson regulator set to the desired pressure and then through a needle valve and a high pressure flex hose to the hydrogen inlet. Air and traces of moisture are removed by coming up to, and then releasing, the pressure two or three times at the beginning of the experiment. The progress of hydrogenation is followed during the process by taking 10-15 g samples at 10-15 min intervals, filtering each to remove catalyst, and determining the decrease in refractive index.

The reactor has been used to follow the complete hydrogenation of a variety of edible fats and oils. Pressures up to 53 psi and temperatures up to 121 C have been used routinely, and the apparatus has been tested up to 200 psi and 130 C. Higher temperatures could be obtained by replacing the 300 watt blanket heater with a higher wattage heater.

Because hydrogen is highly flammable and forms explosive mixtures with air, normal safety precautions should be observed in locating and operating this equipment. Presumably, the reactor could be used to conduct a variety of other specific reactions, such as methylation, hydrolysis, esterification, and interesterification. However, the size of bolts (1.27 cm) is limiting; and, to allow a 5:1 safety factor, the working pressure should not exceed 75 psi. We also recommend that a screw type safety head SF-47, containing

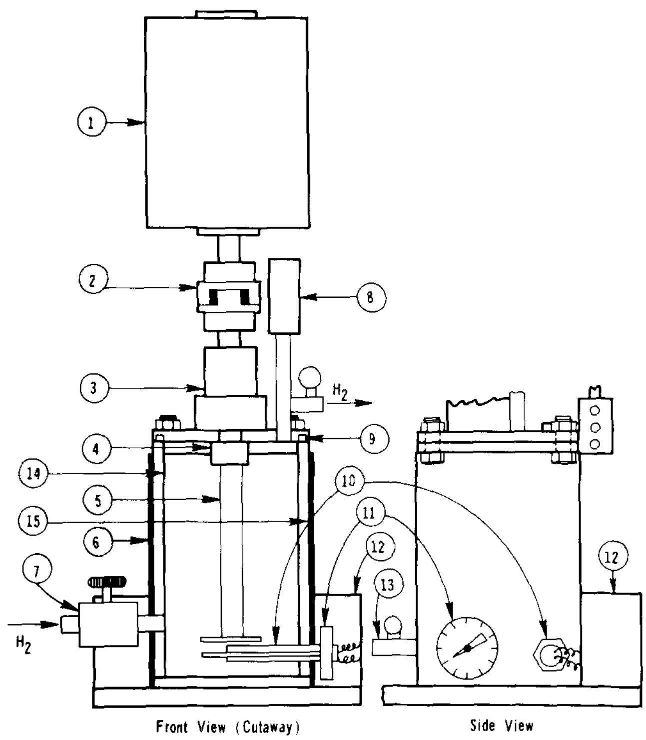


FIG. 1. Schematic of pressure reaction apparatus used for hydrogenation. 1 electric motor, 2 flexible coupling, 3 rotary ceramic seal, 4 stirrer collar, 5 stirrer, 6 heater and insulation, 7 gas (hydrogen) inlet, 8 pressure gage, 9 silicone rubber O-ring, 10 thermoregulator, 11 temperature gage, 12 electrical control box, 13 sample petcock, 14 inside diameter 11.4 cm, 15 outside diameter 12.7 cm.

a 17.4 mm flat seat metal rupture disc type B with a pressure rating of 100 psi at 150 C (BS and B Safety Systems, Tulsa, Okla.) be inserted into the reactor cover in case excess pressure is introduced or generated.

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REFERENCE

1. Smith, L.M., and A. Vasconcellos, JAOCS 51:26 (1974).

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# An Apparatus for the Measurement of Lipoxygenase Activity and Pentane Production[1]

## ABSTRACT

The enzyme lipoxygenase has been demonstrated to produce pentane. However, until now no apparatus has been available to measure the pentane concomitantly with the lipoxygenase activity. Such an apparatus is described by this paper. The apparatus consists of a reaction vessel which can be sealed with a rubber septum to allow sampling for pentane production and an oxygen measuring device to determine the oxygen level of the system. The apparatus is being used to conduct in-depth studies of the lipoxygenase enzyme and its possible role in food quality.

## INTRODUCTION

Pattee, et al., (1) demonstrated that five major volatile compounds were produced during maturation of the peanut seed, with pentane production dominant during the middle and late maturation stages. They postulated that pentane is produced in a reaction sequence starting with the action of lipoxygenase on linoleic acid. To study this further a simple apparatus for the measurement of both lipoxygenase activity and pentane production was developed.

[1]Paper 4128 of the journal series of the North Carolina State University Agricultural Experiment Station, Raleigh, N.C.

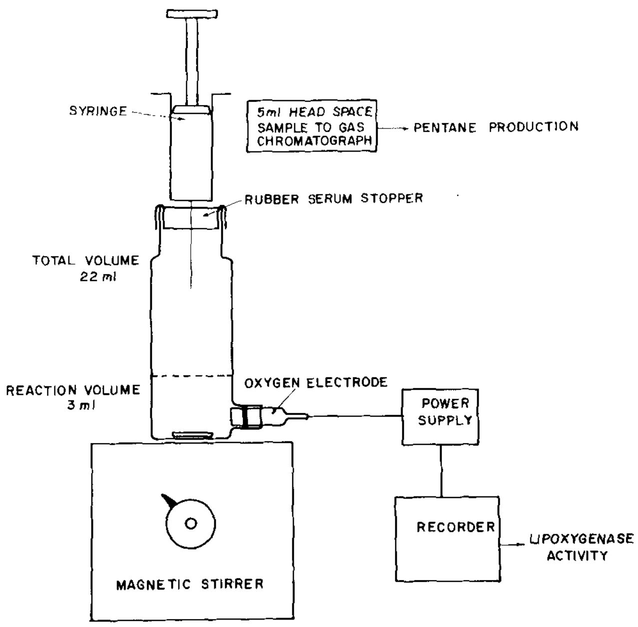


FIG. 1. Apparatus for the measurement of lipoxygenase activity and pentane production.

## EXPERIMENTAL PROCEDURES

Lipoxygenase activity was measured by $O_2$ consumption by the polarographic method of Mitsuda, et al., (2) and pentane production was determined concomitantly at room temperature (25 C ± 2 C) in a glass vessel fitted with a Clark oxygen electrode and a serum stopper (Fig. 1). The vessel

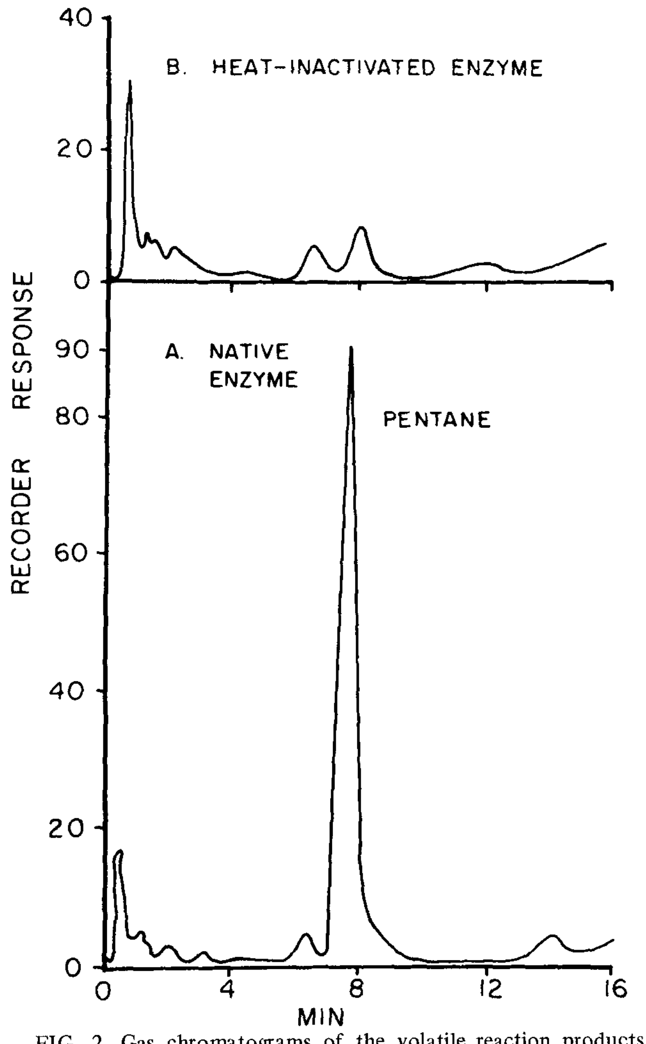


FIG. 2. Gas chromatograms of the volatile reaction products produced from the oxidation of linoleic acid by soybean lipoxygenase; (A) 25 ng crude commercial enzyme and (B) 25 ng heat-inactiviated enzyme.