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MICRO-HYDROGENATION UNIT

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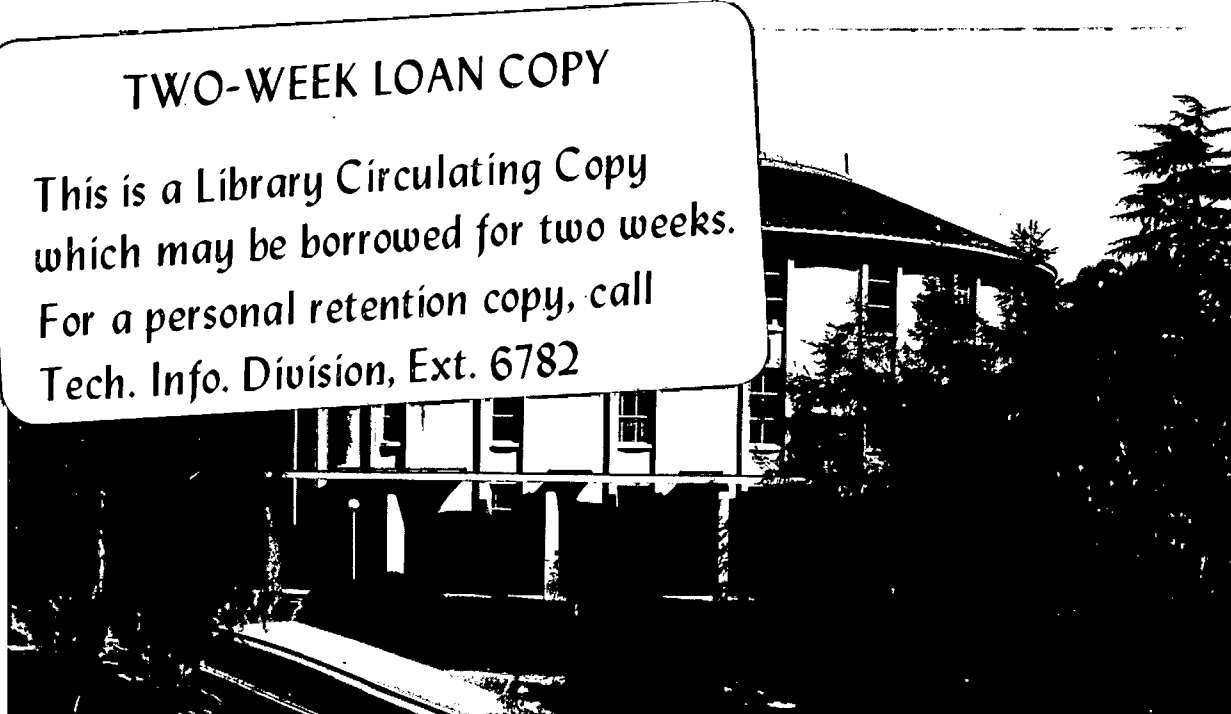
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MICRO-HYDROGENATION UNIT

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During research on the fate of accelerated carbon-14 ions and excited tritium atoms impinging on solid organic targets (1-3) the need for a micro-hydrogenator capable of working with 10 ng-10mg of volatile materials in 100 μ l of solvent became apparent. Commercial units were not suitable and units reported in the literature (4,5) were not readily applicable to our specific needs. The hydrogenation system had to be applicable both to re-injection of fractions isolated from a gas chromatograph and up to 50-100 μ l quantities of sample prepared for further analysis and work-up. Thus on-column hydrogenation for gas chromatography was not considered.

A quite inexpensive (\$35) system was assembled as shown in Fig. 1. The vial (600 μ l Reacti-Vial, Chemical Service, Addison, Ill.) was modified by cutting off 15 mm from the bottom and heating the base until it was slightly rounded. This would allow suspension of the catalyst during agitation. A 1.5 mm hole was drilled in the center of a solid screw cap and the cap gasket replaced by a teflon/silicone liner (Tuf-bond, Pierce Chemical Co.) teflon side down. The needle, a 5 cm x 21 gauge, was inserted through the hole. Tests showed that this arrangement would hold 30 psi H_2 without leaking. The only leaks came from improper seating of the vial lip onto the teflon. The vial lip was then ground flat and a thin film of silicone vacuum grease applied to it. This generally formed an effective seal. The major loss of sample then arises from wetting the catalyst (Pd on C 30% w) and the vial surfaces, a total of 5-10 μ l. This can be further reduced by distilling the sample from one reacti-vial to another via a stainless steel capillary tube.

Assembly of the apparatus is self-explanatory from Fig. 1. Catalyst is added to the vial by dipping a piece of thin walled glass tubing, 1 mm ID, into the bottle of catalyst so that about a 3-5 mm high plug is collected. This weighs approximately 1.5 mg and is blown into the vial. The sample solution is added (50-100 μ l volume) and immediately fastened to the cap-cum-needle assembly and frozen with liquid nitrogen. The system is then alternately evacuated and filled with hydrogen to 30 psi three times to remove the air. After the last filling, the vial is thawed, placed next to the rubber cone of a vortex mixer (Model S-8220, Scientific Products, Inc.) and agitated just enough to keep the catalyst suspended. Thirty minutes at room temperature is adequate for alkyl and cyclic olefins.

After hydrogenation, the sample is again frozen, evacuated, and the vial unscrewed from the cap. A Mini-Inert valve cap (Supleco Inc., Supleco Park, PA) for this size is screwed on, and the sample allowed to thaw. These valves allow one to withdraw part or most of the sample via a hypodermic for injection into the gas chromatograph.

When withdrawing the hydrogenated sample for analysis by GLRC, care must be taken to avoid catalyst pickup by the hypodermic needle which may plug the needle.

Three hydrogenations of cyclohexene in cyclohexane (1:1) μ l were performed with this system to test the completeness of reaction and sample loss. The GLC column was carbowax-20M on Chromosorb W (15%) at 70 C. Integration of the areas was by a Hewlett Packard 3352-B Laboratory data system. The sample weight losses ranged from 2.9% to 3.7%, the product cyclohexane was 99.7% pure.

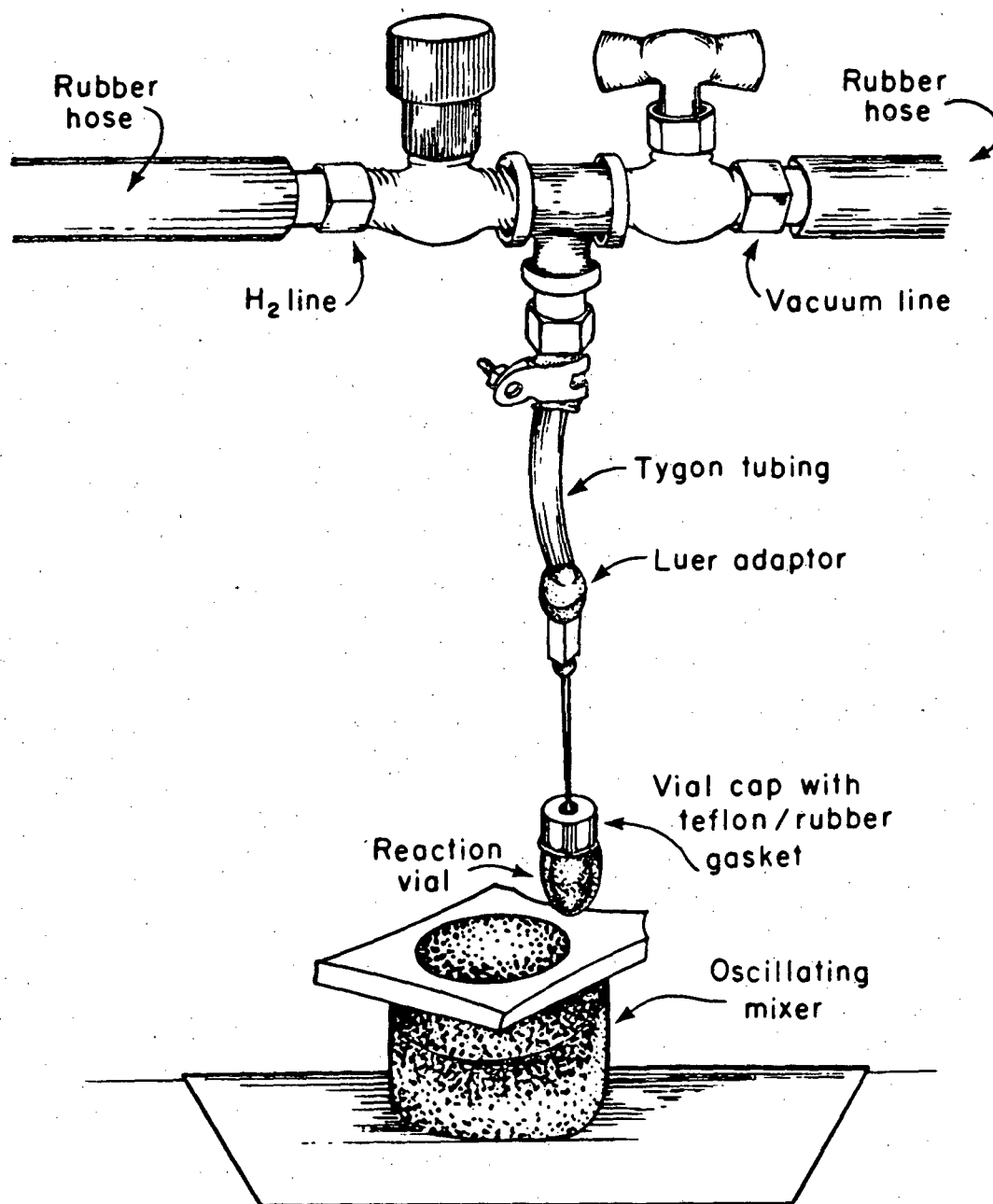
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Micro - Hydrogenation System



XBL 7811 - 4380

Fig. 1.

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