

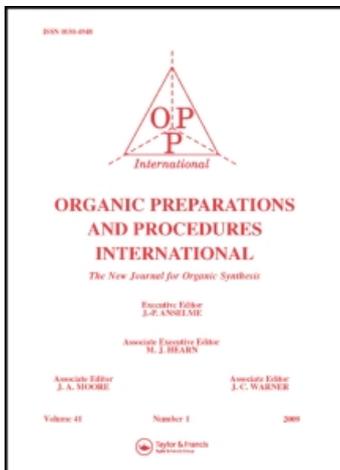
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INEXPENSIVE STEEL REACTION VESSELS AND ACCESSORIES FOR ORGANIC REACTIONS

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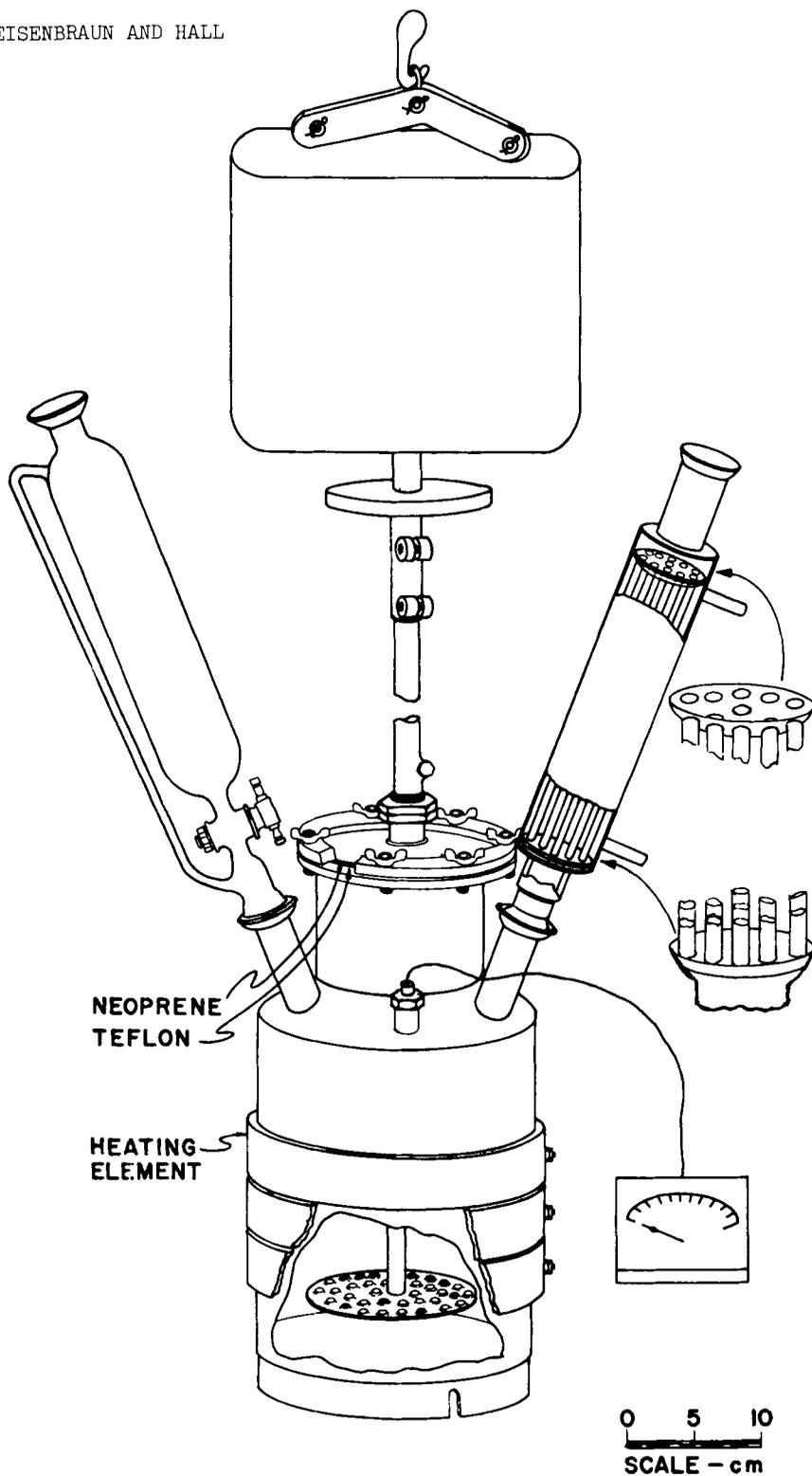
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INEXPENSIVE STEEL REACTION VESSELS AND
ACCESSORIES FOR ORGANIC REACTIONS

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The use of pyrophoric and other hazardous materials in large-scale laboratory synthesis requires safe and effective equipment. Metal reactors are preferable because of the protection they afford against accidental breakage and sudden release of potentially explosive materials. The inexpensive steel vessels described by Hoehn and Johnston² is a major step in overcoming this need since their modification is easy. We were unable to use the vessels as described since stirring was essential. Our modifications provided the 11-1. vessel^{3a} shown in the accompanying figure, which is designed to accommodate an explosion-proof E-2 Vibromixer.^{4a} This change necessitated addition of a ring clamp to secure the gas-tight Teflon and Neoprene rubber sandwich which serves as a diaphragm.^{4b} A 22-1. vessel^{3b} of similar design was also constructed and its operation was equally satisfactory. Our modifications included installation of two 35/25 stainless steel sockets⁵ on the shoulders of the vessels. These serve as ports for an addition funnel and a reflux condenser. Cooling is accomplished by immersing the vessel in water, ice-salt or Dry Ice. Heating is done with a water bath, added circular strip heaters⁶ or a blanket heating mantle.⁷ The construction of the multitube, stainless steel condenser completed the apparatus and reduced the hazard associated with accidental introduction of water from a cracked glass condenser.

We have used the described apparatus effectively in reactions involving several types of metal hydrides, aluminum alkyls, Grignard reagents,



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hydrazine and alkali (Wolff-Kishner),⁸ and alkaline hydrolysis. The use
of acidic reagents which may attack the vessels is avoided.

Hoehn and Johnston² state that installation of a sight-glass did not
solve the problem of viewing the interior of the vessel. We have no good
solution to this design problem, but we have successfully monitored the
temperature with a thermocouple and pyrometer.⁹ Reflux may be observed
by introducing a glass adapter, fitted with an O-ring joint,¹⁰ between
the steel condenser and the steel vessel.

We thank the Research Foundation, Oklahoma State University, for
support of this work.

REFERENCES

- (1) Manager, Chemistry-Physics Shops, Oklahoma State University.
- (2) W. M. Hoehn and J. D. Johnston, *J. Chem. Ed.*, **43**, 537 (1966).
- (3) (a) Container, stainless steel, 2.5-gal. Firestone 27009.
(b) Container, stainless steel, 5-gal., Firestone 26929, Firestone
Steel Products Company, Akron, Ohio 44301.
- (4) (a) Chemapec, Inc., Hoboken, N. J. 07030.
(b) The 1/4-in. Neoprene rubber diaphragm, supplied by Chemapec, Inc.
is attacked by some boiling reactants and solvents. We corrected this
by adding a 1/64-in. layer of Teflon on the reaction side of the
diaphragm.
- (5) Available from Kontes Glass Co., Vineland, N. J. 08360.
- (6) Strip heaters, 750 watts, 1.5-in. width, Type SE, Monel sheath, 25.5-
in. length, curved into a complete circle with ends touching and
terminals outside of curvature, for 11-l. vessel. Similar 1000-watt
strip heaters 38.5 inches in length are used with the 22-l. vessel.
E.L. Weigand Co., Pittsburgh, Pa. 15208.
- (7) Glas-Col Apparatus Co., Terre Haute, Ind: 47802.
- (8) E. J. Eisenbraun and H. Hall, *Chem. Ind. (London)*, 1535 (1970).
- (9) Available from Assembly Products, Inc., Chesterland, Ohio 44026.
- (10) An O-ring joint available from West Glass Corp., South El Monte,
Calif. 91731, is essential to overcome unequal expansion or contrac-
tion of glass and metal. The dropping funnel should be fitted with
an O-ring joint.

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