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Rotating disk electrodes are widely used in electrochemical research since their hydrodynamics and mass transfer characteristics are well understood.[1] Several modifications of experimental rotating disk electrode assemblies have been described.[2,3] The present design differs from previous ones in that it allows the use of a rotating disk electrode under inert atmosphere conditions. The apparatus is thus well suited for the study of electrode reactions under controlled mass transfer conditions when the electrolyte and/or electrode is unstable in the presence of air or moisture.

The described apparatus consists of two parts. (1) A rigidly mounted drive unit contains the motor and the electrical contact to the rotating axle. (2) A detachable cell unit consists of the electrolytic cell with the rotating disk working electrode, and of a special gas seal arrangement with an argon bypass. The particular design of the apparatus allows the cell unit to be transported to a glove box for filling operations, while electrochemical measurements are performed outside the box under exclusion of air and moisture.

A commercially available torque unit* containing a DC motor with closed loop tachometer feedback allows continuous variation of the

* SERVO-TEK Model ST-573-1, Servo-Tek Products Co., Inc., Hawthorne, N.J.

rotation speed between 20 and 3600 rpm. The angular velocity is constant within 1/2%. Calibration of the system was done stroboscopically against 60 cps line frequency harmonics or, at the lowest rotation speeds, by means of a stopwatch. The shaft of the drive unit is rigidly fixed to the upper part of the hollow axle. Electrical contact to the axle is made by means of a stainless steel cylinder rotating in a mercury pool. A mechanical coupling with rubber insert containing plugs for electrical contact connects the drive unit to the removable cell unit. A small spring in the female plug provides for slight flexibility of the contact. The cell unit is shown in Fig. 1. It is positioned with respect to an aluminum support by means of three set screws attached to the stainless steel cell cover. The lower part of the axle, which is part of the detachable cell unit can be raised or lowered slightly with respect to the cell cover by turning a brass bearing unit threaded onto the argon bypass. Figure 2 shows the detailed arrangement. A "teflon" O-ring is pressed against a protruding metal ring when the axle is in the upper position, thus sealing the cell. In this arrangement the cell unit can be transported from the dry box to the drive unit. Before an electrochemical experiment is started the argon bypass is flushed with argon. The shaft is then lowered in order to allow free rotation during the experiment. Backdiffusion of oxygen is excluded by passing the argon through an oil filled flask on leaving the apparatus. Argon is flowing through the ante chamber throughout the measurements. Rotating disk working electrodes are made of stainless steel or nickel press fitted into a teflon insulation. The electrode diameter is 1/8 inch, the total disk diameter 1/2 inch. The electrodes are screwed onto the axle and

positioned as indicated in Fig. 2. Eccentricity of the electrodes is less than 0.2 mil.

A glass cell made from industrial Pyrex glass pipe is attached to the stainless steel cell cover by means of a conventional cast aluminum flange* and sealed with a teflon O-ring. The cell volume is kept small because of the lengthy electrolyte purification procedures required when working with nonaqueous solvents. 250 ml solution are needed per experiment. A platinum wire melted into the glass wall serves as electrical contact to the counter electrode, either an amalgam electrode or a platinum sheet. Rigidly attached to the glass cell is the compartment for the reference electrode. A capillary in the cell wall provides electrolytic connection between cell and reference electrode. The capillary is positioned in a horizontal plane with the disk electrode at virtually infinite distance. This arrangement allows easy correction of IR drop included in the measured electrode potential.[4] The particular construction of the electrolytic cell using standard pyrex glass pipes and fittings allows convenient interchange of glass cell types, e.g., a cell of larger volume or a U shaped cell may also be used.

The argon supply is connected to the argon inlet and the reference compartment by means of a three way stopcock. Thus pressure differences between cell and reference electrode compartment are avoided. Constant temperature is maintained during the experiments by inserting the cell into a regulated temperature bath (±0.1°C) resting on a platform which can be raised and lowered.

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* Pyrex Conical Process Piping System, Corning Glass Works, Building Products Department, Corning, New York.

ACKNOWLEDGEMENT

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REFERENCES

1. V. G. Levich, Physicochemical Hydrodynamics (Prentice Hall Inc., New York, 1962).

2. A. C. Riddiford, Advances of Electrochemistry and Electrochemical Engineering, Vol. IV , Paul Delahey and Charles W. Tobias, editors, (Interscience Publishers, New York, 1966), pgs. 47-117.

3. H. E. Hintermann and E. Suter, Rev. Sci. Instr. 36, 1610 (1965).

4. John Newman, J. Electrochem. Soc. 113, 501 (1966).

FIGURE CAPTIONS

Fig. 1 Detachable cell unit.

- b mechanical coupling
- c set screws
- d reference electrode
- e electrical contact
- j threaded brass bearing unit
- k stainless steel cell cover
- l argon inlet

Fig. 2 Cross-section of argon bypass and seal.

- a bearings
- f teflon O-ring for sealing cell during transportation between dry box and drive unit
- g rotating disk electrode embedded in teflon (t)
- j threaded brass bearing unit for raising and lowering of shaft
- k stainless steel cell cover
- l,m argon bypass
- n hollow shaft
- x teflon washer
- y isolation
- z threaded bakelite insert

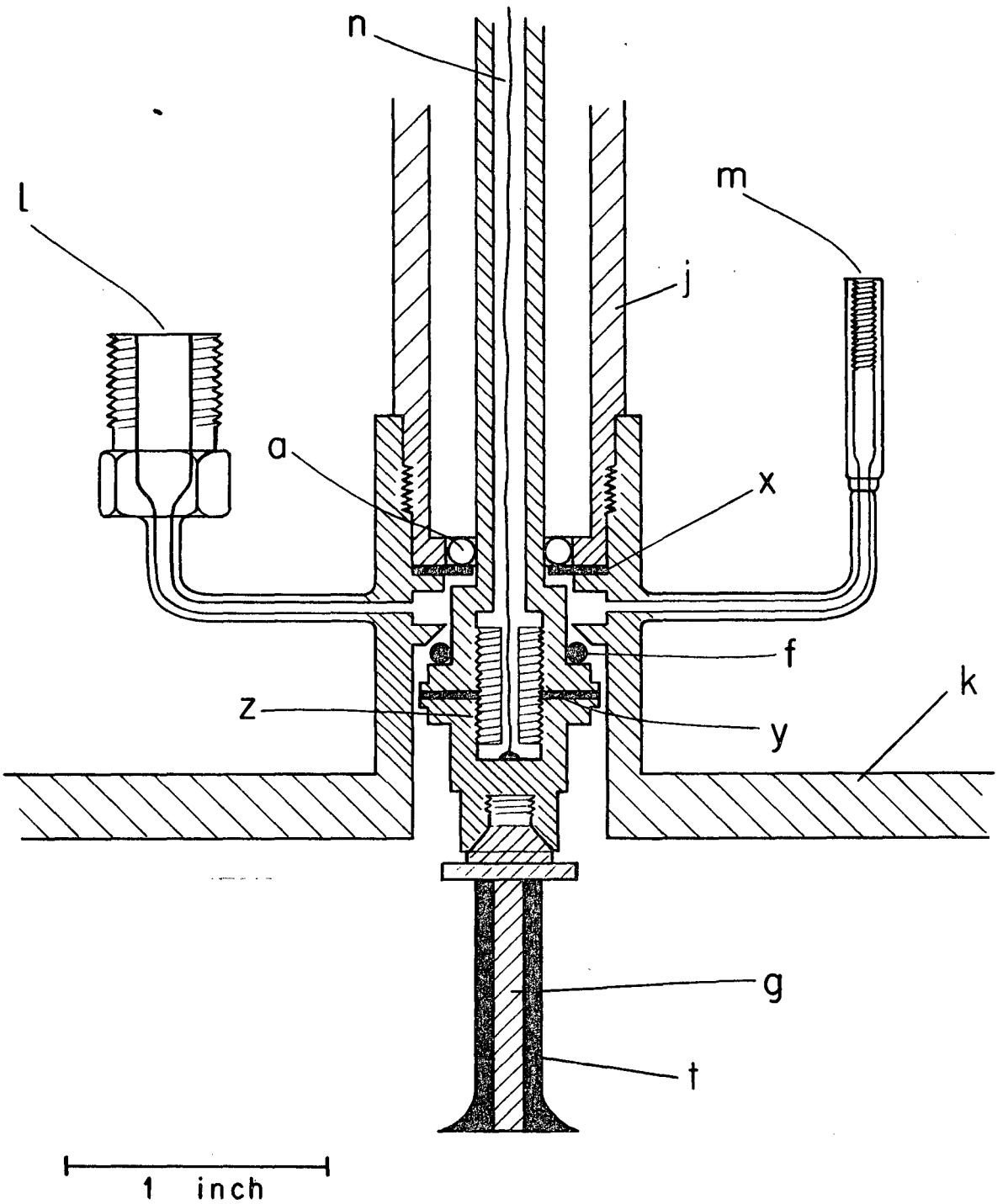

Fig. 1

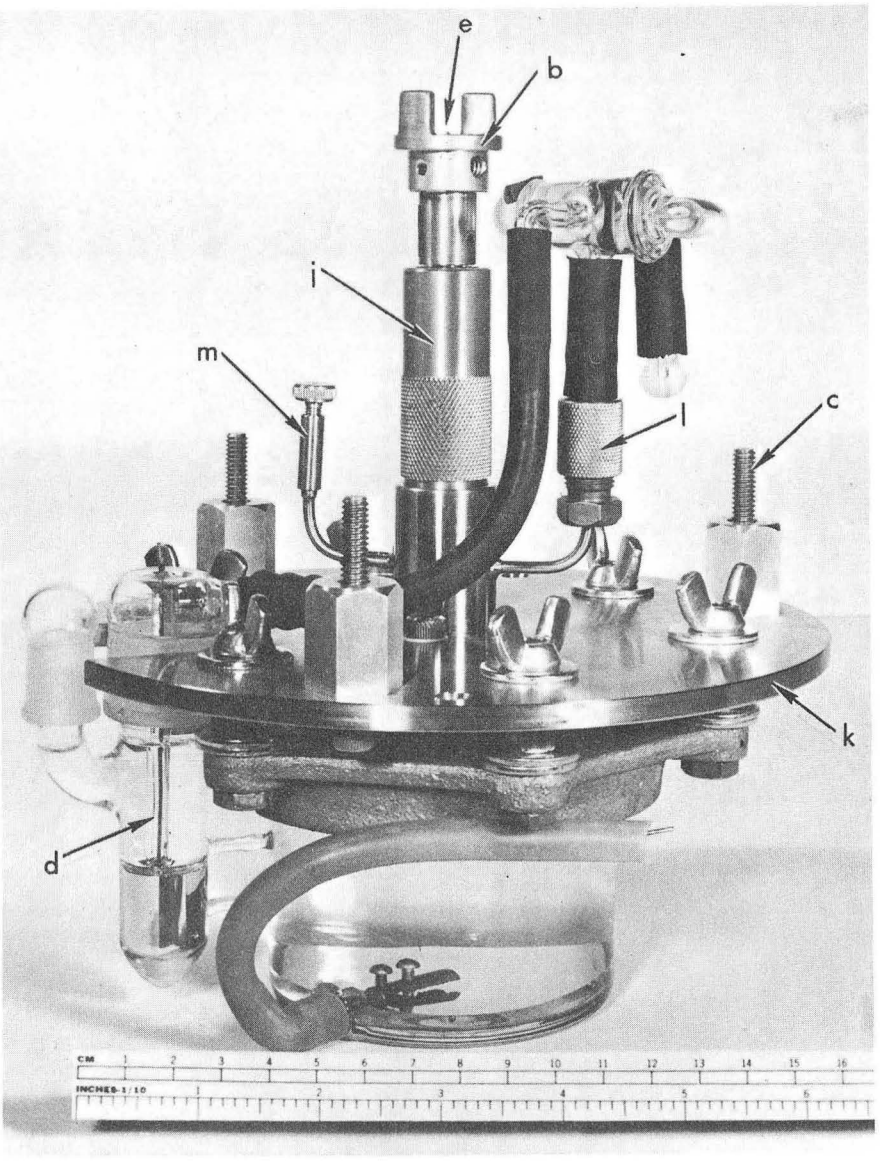

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Fig. 2