An improved glove box with high purity atmosphere

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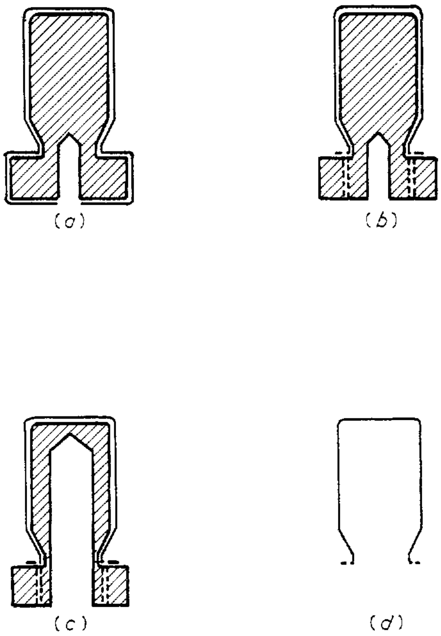

Figure 2 (*a*) Former leaves bath with complete copper plate; (*b*) holes drilled in flange, unwanted copper machined away; (*c*) bulk of former drilled out, remaining material removed by immersion in hot caustic; (*d*) finished calorimeter shell, approximately 0·009 in thick

removing the latter by the application of heat, leaving the inside walls of the tubes conveniently tinned. Copper tubes can also be formed as integral parts of a component by plating on aluminium pins inserted into sockets in the body of the former. Uniform plating is greatly facilitated by painting around the joints with silver paint (Johnson Matthey Metals Ltd), which has the advantage of providing good electrical continuity and also a curved surface on which to electroplate.

3 The plating process

Some considerable success has been achieved using Sulfast Bright Acid copper plating solution (Canning 1966, p. 444). The method used has been to clean the former thoroughly in an ultrasonic bath, rinse with running water, immerse in a 10% sulphuric acid solution, rinse again, and put into the plating bath. Plating is commenced at a low current density and the current gradually raised to the required value over a period of approximately 15 min. The plating bath in use at present was purchased from W Canning and Co. Ltd, and is 14 in^3, rubber lined, and has a reciprocating motor for slow agitation of the cathode. Before this, quite satisfactory results were obtained in a large beaker equipped with a stirrer.

For the sake of completeness rather than from any dissatisfaction with the above process, Cuprax (Canning 1966, p. 433) plating solution allied to the Bondal process (Canning 1966, p. 272) for plating on aluminium was tried. This should be a superior method since it has a better ability to cover irregular shapes uniformly. In practice the technique has not been found successful because the deposit is susceptible to blisters, formed by pockets of gas trapped beneath the copper plate.

4 Conclusion

The methods outlined here are of common electroforming practice and as such are not new. The purpose of this note has been to illustrate that this technique is worth considering when relatively simple forms are required, and that satisfactory results can be obtained with unsophisticated equipment, and with only a little practice.

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An improved glove box with high purity atmosphere

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Abstract Modifications and performance are given for a glove box with high purity atmosphere by absorption of H_2O, O_2, N_2 and other impurities.

An 'economical' glove box described earlier (Gardner 1964) was operated successfully over several years and this note describes modifications and reconstruction of the purification train to reduce breakage and increase capacity. Detailed drawings and explanatory notes of the new system which is made almost wholly of metal are available upon application to the author. The flow diagram of the system is given in figure 1.

Modifications to the original design are (i) replacement of type 4A molecular sieves by type 13X to facilitate removal of organic vapours as well as moisture; (ii) replacement of manganous oxide by BTS copper catalyst (supplied by BASF, Ludwigshafen am Rhein), in order to conduct regeneration

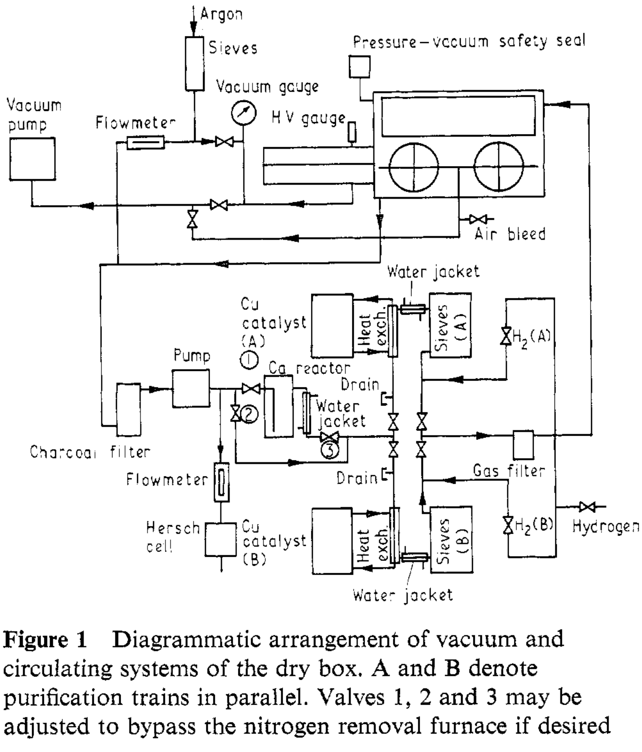

Figure 1 Diagrammatic arrangement of vacuum and circulating systems of the dry box. A and B denote purification trains in parallel. Valves 1, 2 and 3 may be adjusted to bypass the nitrogen removal furnace if desired

at 150° instead of 350° and permit a large number of regeneration cycles; (iii) addition of a calcium reactor at 650° for removal of nitrogen; (iv) addition of an activated charcoal tower to remove various impurities and decomposition products; (v) addition of a Norgren type 30BE-4(25) gas filter to reduce entry of dust into the dry box; (vi) addition of a small entry port operated by flushing with inert atmosphere to enable the rapid transfer of weighing bottles, spectra cells and other small objects; (vii) continuous metering of the oxygen content by a Hersch cell of modified design (Morgan 1963). (Since the oxygen content was usually found to be greater than the moisture content a low oxygen was considered to be a satisfactory criterion of the purity of the atmopshere of the box.) The increased capacity of the oxygen removal system was such that an oxygen content of 1% was lowered to 10 p.p.m. in less than 30 min. The concentration of 1% oxygen was achieved either by the plastic bag displacement technique (Gardner 1964) or by flushing the box, initially containing air, with 1 l min^{-1} of pure argon overnight.

With an overnight argon purge of only 10 ml min^{-1} to maintain a positive pressure within the box and storage of the gloves (initially under vacuum) within the evacuable glove ports, the lapse of time in the morning before working levels of purity were achieved was limited to the response time of the Hersch cell.

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A simple x-ray crystal monochromator

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Abstract A small, simple x-ray plane-crystal monochromator used with a horizontally mounted x-ray tube is described. The monochromator is designed to produce a high intensity beam of characteristic radiation in the wavelength range from CrKα to MoKα.

The following simple monochromator design has been found to be useful and convenient for work with a Buerger precession camera. It could also be used with other x-ray diffraction cameras.

The monochromator (shown in figure 1) consists of a brass base plate A, 1½ in long and 1½ in wide, with an adjustable dovetail on one face and a right-angle section, slotted to take a knurled captive screw B. This screw is tapped into the male half of the dovetail C and provides a smooth horizontal translation of about 20 mm for the monochromator housing which is attached to C. The entrance side and base are made from a section of right-angle brass, ensuring that the floor is perpendicular to the base plate A. Rigidly attached to the right-angle section is the curved exit side containing an elongated slit and a recess to accept a similarly curved slide D.

A number of these slides with exit ports E at the appropriate positions for CrKα, CuKα and MoKα, were constructed. The slides can be set and locked using the locking screw F

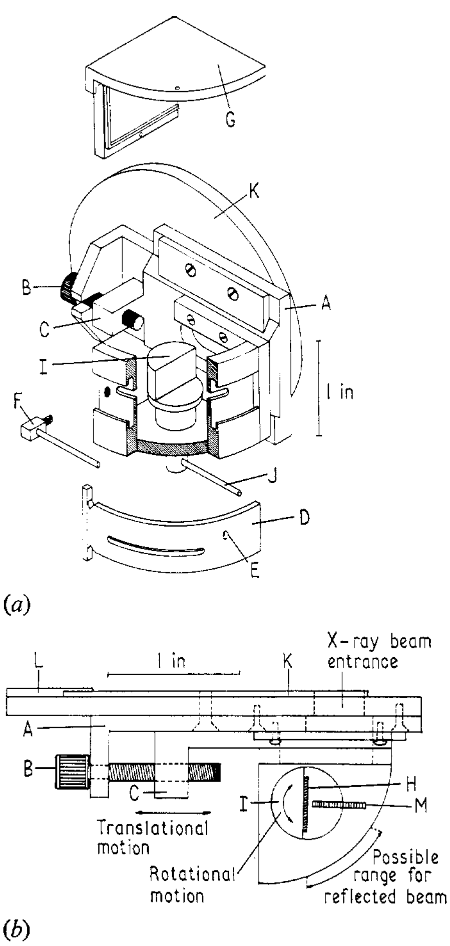

Figure 1 (*a*) Exploded view of the monochromator housing. (*b*) Schematic plan of the monochromator: A, base plate with dovetail; B, captive screw to control translation; C, male part of dovetail; D, curved slide; E, exit port; F, locking screw for slide; G, top cover; H, monochromating crystal; I, crystal holder; J, lever to control rotation; K, adaptor plate; L, locating section on adaptor plate; M, baffle to prevent leakage

(see figure 1(*a*)). Two screws secure the cover G to the housing. Easy removal of this section gives access to the interior. Leakage of stray radiation is prevented by overlapping the fitted sections.

The plane monochromator crystal H is mounted on a simple holder I, the shank of which is inserted into a precisely machined hole in the floor of the housing. A lever J through the bottom of the shank is used to rotate the holder about a vertical axis. The holder is held in position with a friction lock provided by a spring and washer on the shank above the lever (not shown in figure 1). Rotation of the crystal controls the angle at which the x-ray beam strikes it, providing a useful range of Bragg angles between 0 and 70°. A large range of wavelengths can thus be obtained from one crystal. The crystal can also be readily replaced by another one mounted on a spare crystal holder, the only critical requirement in positioning the crystal being that it be parallel to the flat, vertical surface of the holder.

The monochromator was initially fitted directly on the high power tube of a Hilger-Watts Y40 x-ray generator, and tested for ease of operation and efficiency. A simple adaptor plate K, replacing the normal shutter plate, enabled us to transfer it to the point-source port of a standard horizontal