

Short Communications

An apparatus for the equilibration of columns prior to use in vapor phase chromatography*

In an effort to separate many diverse types of compounds by vapor phase chromatography, it became necessary to evaluate a large number of potentially useful column packing materials. The equilibration of the packed columns while connected to the chromatographic instrument** precluded its analytical use and occasionally fouled the conductivity cell. Hence, other means of conditioning the columns were sought. The usual practice of conditioning the columns independently (merely placing the columns in an insulated rack and heating electrically while nitrogen flow is maintained) was found to be unsatisfactory because of the high rate of convection cooling and the

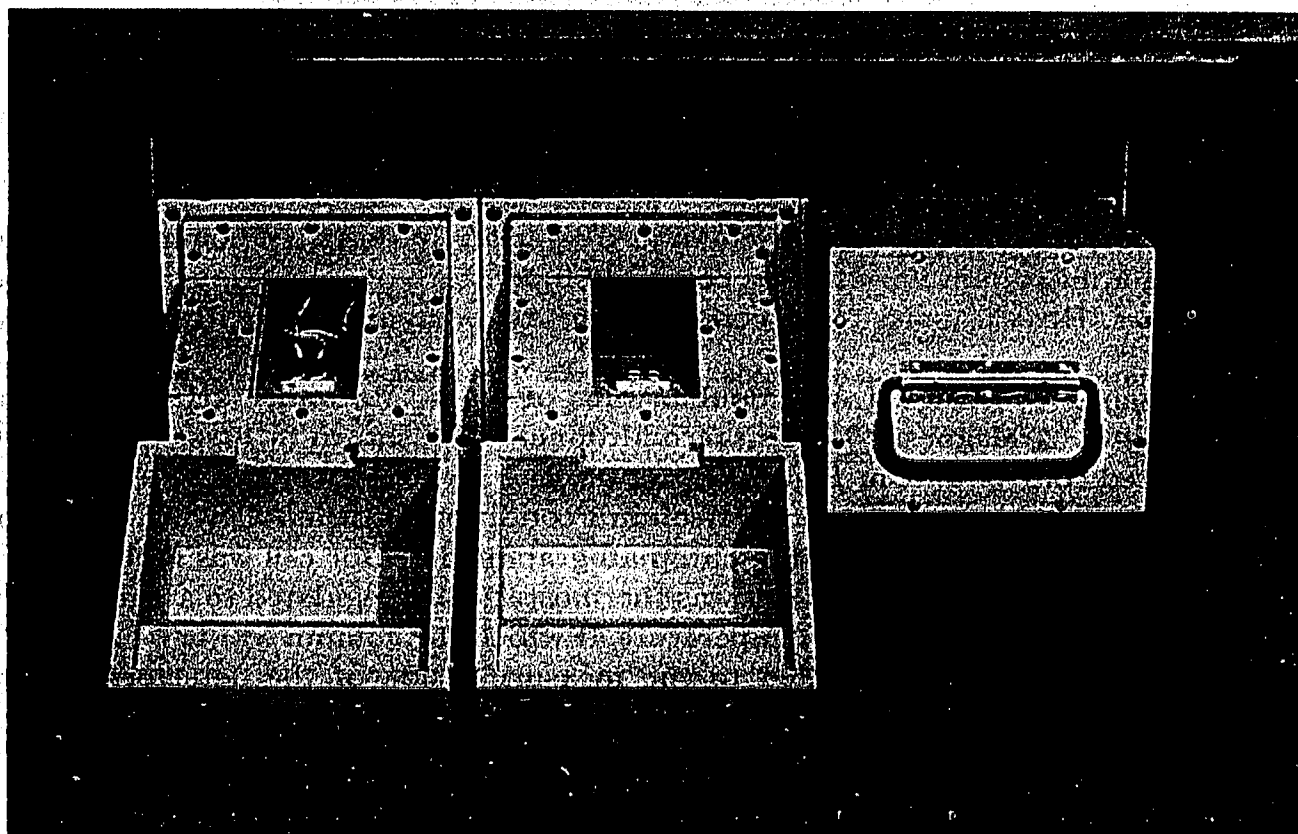


Fig. 1. 3-column preconditioner (end view).

* This work was supported by Research Contract No. Saph 70155 with the Air Pollution Medical Program, Public Health Service, Department of Health, Education, and Welfare.

** Burrell Kromo-Tog: Model K-2.

presence of uneven heating. For these reasons, a device was designed specifically for the conditioning of columns. With a minimum of conversion it may also be used as a three-unit chromatographic instrument.

Apparatus. The preconditioner built in this laboratory is essentially a well insulated chamber with provision for the flow of heated gas through the column while it is held at some predetermined temperature. This particular unit is designed to service 3 Burrell hairpin type columns of any length up to 2.5 m with individual control of gas flow and heater voltage. It is capable of maintaining temperatures in

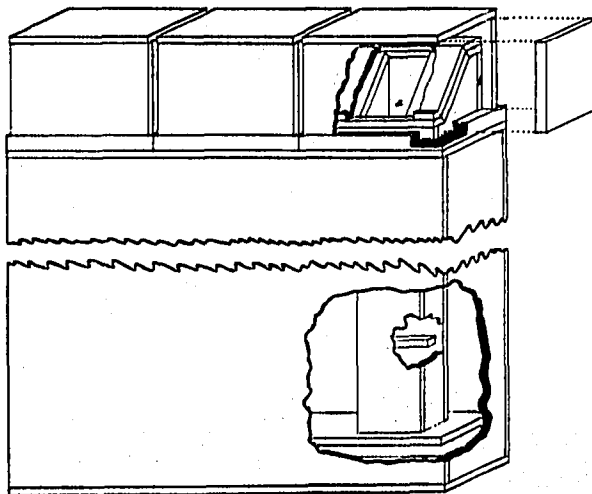


Fig. 2. 3-column preconditioner (cut-away view).

excess of 400° , shows a maximum temperature variation along the column length of $\pm 0.5^{\circ}$ and a maximum variation in column temperature of $\pm 1.0^{\circ}$ over a period of several hours. Heat transfer from compartment to compartment within the unit is negligible.

The oven is constructed with double walls of $\frac{1}{2}$ in. Maronite* separated by at least 1 in. of 85 % Magnesia**. The preconditioner*** is shown in Figs. 1 and 2 and the associated control unit in Fig. 3.

Methods. To obtain a criterion of column conditioning, the following procedure was set up: The reference side of the chromatographic detector cell was replaced by a fixed resistance, and the detector cell was thermostatically regulated to a temperature at least as high as the maximum temperature at which the column was to be used. The column in question was connected to the measuring side of the detector cell and flushed with inert gas at room temperature until a stable base line was obtained.

After attainment of a stable base line with the column at room temperature, the column temperature was gradually raised until upward deflection of the base line occurred.

* Maronite, Johns Manville Co.: A sheet product composed of asbestos fiber and diatomaceous earth with an inorganic filler.

** 85 % Magnesia, Johns Manville Co.: A coarse powder containing 15 % asbestos fiber.

*** Blueprints can be made available on request.

The temperature at which the base line showed a deflection of 0.5 % full scale at maximum sensitivity was considered to be the maximum allowable operating temperature (MAOT) for each column after the particular conditioning applied.

Results and discussion. Fig. 4 indicates the manner in which conditioning time affected the MAOT for a pair of typical columns. The shape of the curve appears to be

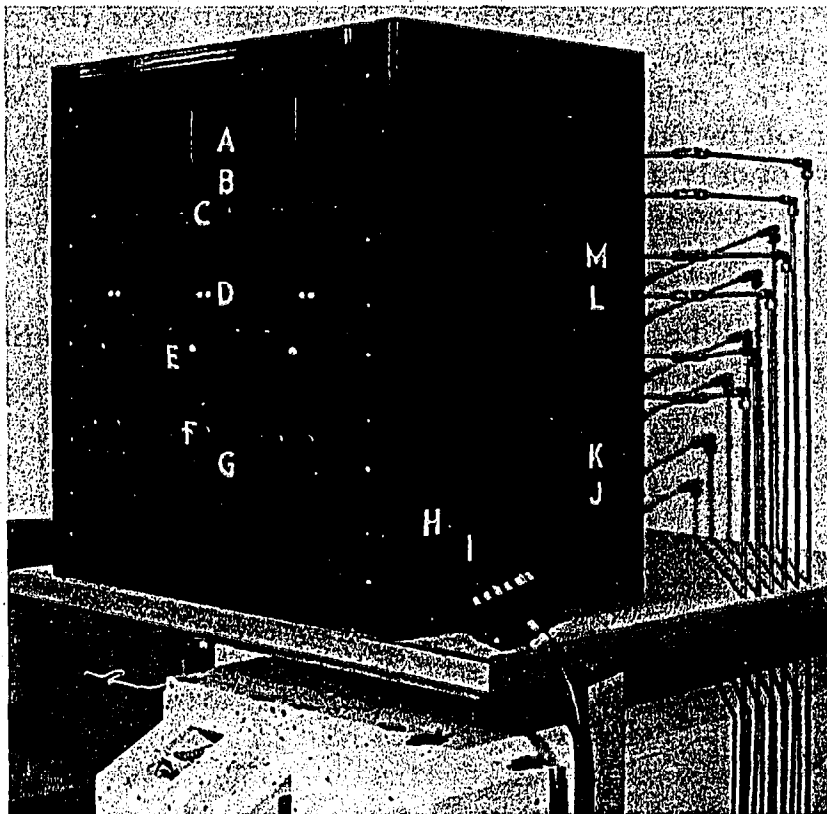


Fig. 3. 3-column preconditioner control unit. A, flow meter; B, needle valve; C, input and output toggle valves; D, thermistor outlets; E, powerstat; F, switch, fuse and pilot light cluster; G, auxiliary powerstat outlet; H, cable connector, preconditioner to control unit; I, main power inlet; J, gas inlet, from tank; K, gas outlet, to atmosphere; L, gas stream, to oven No. 2; M, gas stream, from oven No. 2.

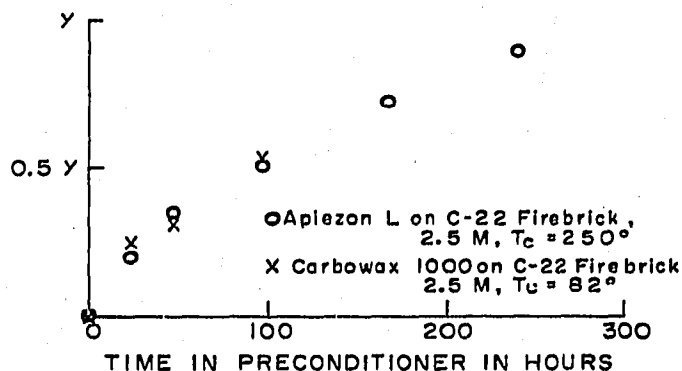


Fig. 4. Plot of Y as a function of time for a constant preconditioner temperature where: $Y = \frac{T - 20}{T_c}$; T = maximum allowable operating temperature, and T_c = preconditioner temperature.

sufficiently regular to permit the prediction of required conditioning times on this basis.

The Apiezon L column was maintained at a temperature of 250° in the preconditioner until the desired MAOT of 245° was attained. For this column 4 days of conditioning per m column length were required. This conditioning time is far in excess of that usually considered adequate, and yet is absolutely essential for satisfactory temperature-programmed operation.

Temperature-programmed operation of this column over the range 0–240° has resulted in a base line deflection of less than 0.5 % full scale at maximum sensitivity.

In the case of the Carbowax 1000 column shown in Fig. 4, an MAOT of 45° was desired. Operation of the preconditioner at 82° allowed us to obtain this MAOT with a time expenditure of only 2 days per m column length. This illustrates the somewhat evident point that the conditioning time for a column may be significantly reduced by operation of the preconditioner at a temperature as far as possible in excess of the MAOT desired.

Further studies will be required to determine the effect of conditioning time and rate on the height equivalent theoretical plate.

*Department of Biochemistry, Baylor University College
of Medicine, Houston, Tex. (U.S.A.)*

J. A. COOPER*
R. CANTER
F. L. ESTES
JOSEPH H. GAST

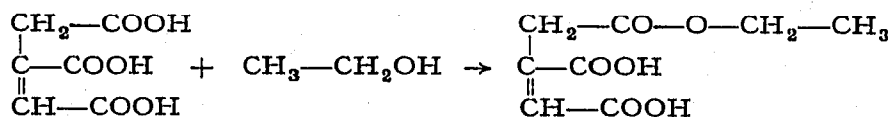
Received May 22nd, 1959

J. Chromatog., 3 (1960) 87–90

* Public Health Service Research Fellow of the National Heart Institute.

Dédoublément de la tache d'acide aconitique par estérification

Dans les études chromatographiques d'acides organiques, il est une tache maintes fois signalée: JERMSTAD ET JENSEN¹, par exemple, l'ont trouvée parmi les acides organiques de l'Aconit et l'ont attribuée à quelque acide inconnu. Nous avons retrouvé cette tache dans l'Aconit, dans le Blé, etc. Son R_F est nettement plus grand que celui de l'acide aconitique, surtout en milieu alcalin; il se situe même légèrement au delà de l'acide fumarique. Dans le tableau des R_F , ou plus exactement des R_G (Glycolique), des acides organiques que nous avons publié², tandis que l'acide aconitique va à 20 en milieu alcalin et à 135 en milieu acide, cette tache se situe à 75 en milieu alcalin et 155 en milieu acide: il s'agit de l' α -monoaconitate d'éthyle



Après extraction alcoolique faite sur des tissus où se trouve de l'acide aconitique, il apparaît régulièrement en assez grande abondance. L'alcool éthylique étant le