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Note

Frit profiles for packed chromatographic column terminations

MARCEL J. E. GOLAY
The Perkin-Elmer Corporation, Norwalk, CT (U.S.A.)
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Special arrangements for the uniform distribution of samples near the entrance of a packed column and, likewise, for their uniform collection can serve to reduce band spreading. For this purpose retentionless frits with suitable profiles have been developed. The use of such frits is particularly pertinent in large diameter columns, *e.g.*, $\geqslant 3$ mm I.D.

Figs. 1 and 2 illustrate two column cross-sections with frit profiles calculated by use of the theory outlined below, the first designed for a coarse particle packing and the second for finer particles. The column packing is shown as large squares and the frit as small ones. The parameters are as follows:

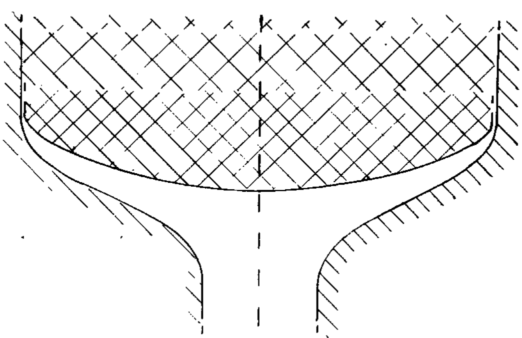

Fig. 1. Frit profile for a coarse particle packing: $s/r_0 = 0.01$.

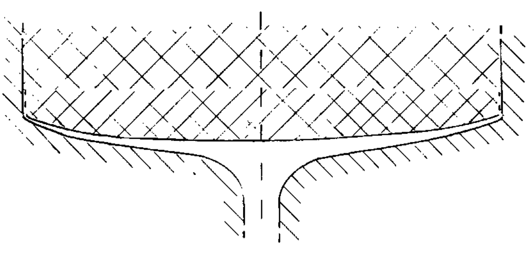

Fig. 2. Frit profile for a fine particle packing: $s/r_0 = 0.001$.

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r_0 = inside column radius
r_1 = entrance and exit pipe radii
r = distance from column axis
t = thickness of the free interspace between the frit and the column wall
t_0 = initial thickness of the interspace at the inner wall of the column exit
V_0 = average fluid velocity within the column
V = average fluid velocity in the interspace
α = angle between the free interspace flow direction and the column axis
p'_0 = vertical pressure gradient within the frit
p'_1 = pressure gradient in the direction of flow in the interspace
s = characteristic dimension of the frit particles
μ = fluid viscosity
x = abscissa normal to the column axis
y = ordinate along the column axis

The characteristic dimension, s, of the frit packing is defined as the quantity which permits the average fluid velocity, v_0, within the column and within the frit to be written as

$$v_0 = p'_0 s/\mu \tag{1}$$

The order of magnitude of s will be one tenth the frit or packing particle diameter.

The fulfillment of four conditions is required to insure the uniform distribution and collection of the fluid, and these conditions can be stated, without loss of generality, in terms of exit conditions. First, the simultaneous arrival of all fluid elements at the collecting point demands that the average fluid velocity within the interspace has a vertical component equal to the average fluid velocity:

$$v \cos \alpha = v_0 \tag{2}$$

Secondly, the average velocity at any distance, r, from the axis, when multiplied by the interspace thickness, t, at that distance and by the circumferencial length at that point should equal the total fluid flow collected between that circumference and the column wall, *i.e.*

$$2\pi rtv = \frac{\pi\,(r_0^2 - r^2)}{2\,rt} \cdot v_0$$

or

$$v = \frac{r_0^2 - r^2}{2\,rt} \cdot v_0 \tag{3}$$

Thirdly, the condition that isobars within the column packing and within the frit be planes normal to the column axis requires that the pressure drop within the interspace, when multiplied by cos α, be equal to the vertical pressure drop within the column:

$$p'_1 = p'_0 \cos \alpha \tag{4}$$

Fourthly, the pressure drop within the interspace should be such as to produce within the interspace the velocity given by eqn. 3:

$$v = \frac{t^2}{12\,\mu}\cdot p_1' \tag{5}$$

The essential task of obtaining the frit profile is now algebraic. From eqns. 4 and 5, and then with 1, we obtain

$$v = \frac{t^2}{12\,\mu}\cdot p_0' \cos\alpha = \frac{t^2\,v_0}{12\,s^2}\cdot\cos\alpha$$

and, with eqns. 2 and 3:

$$\frac{v}{v_0} = \frac{t^2}{12\,s^2}\cdot\cos\alpha = \frac{1}{\cos\alpha} = \frac{r_0^2 - r^2}{2\,rt} \tag{6}$$

Elimination of cos α from eqn. 6 gives

$$\frac{t^2}{12\,s^2} = \frac{(r_0^2 - r^2)^2}{4\,r^2t^2}$$

whereby

$$t = \sqrt{\frac{\sqrt{3}\,(r_0^2 - r^2)s}{r}} \tag{7}$$

and again with eqn. 6:

$$\cos\alpha = 2\sqrt{\frac{\sqrt{3}\,rs}{r_0^2 - r^2}} \tag{8}$$

Eqns. 7 and 8 determine the essential portions of the frit profile. When cos α approaches unity near the column inner wall, the behavior of this profile can be determined more conveniently by writing

$$r = r_0 - 2\sqrt{3}\,s - x \tag{9}$$

from which we obtain

$$\cos\alpha \approx \sqrt{1 - \frac{x}{2\sqrt{3}s}} \approx 1 - \frac{\alpha^2}{2} \tag{10}$$

or squaring and writing

$$\alpha \approx \mathrm{d}x/\mathrm{d}y$$

we obtain

$$\frac{\mathrm{d}x}{\mathrm{d}y} = \sqrt{\frac{x}{2\sqrt{3}s}}$$

$$\mathrm{d}y = \sqrt{\frac{x}{2\sqrt{3}s}}\,\mathrm{d}x$$

$$y = \sqrt{8\sqrt{3}sx}$$

and finally:

$$x = y^2/8\sqrt{3}s \tag{11}$$

This indicates that the theoretical frit profile ends abruptly at the base of a parabola with the radius of curvature $4\sqrt{3}s$ at that point, which is at a distance $2\sqrt{3}s$ from the inner wall.

Figs. 1 and 2 show the frit profiles for the two cases $s/r_0 = 0.01$ and 0.001, as well as the associated profiles of the steel tube of the column at a distance t from the frit. The part of these two figures which is of interest is the intermediate portion from near the exit (or entrance) tube to near the end of the column straight wall. As shown it represents a physical impossibility near the column exit because supporting ribs must be provided to hold back the frit and the column packing; it is nevertheless an ideal solution which the column designer could strive to approximate.

Near the center, the steel tube profile is shown smoothly joined to an entrance or exit pipe calculated to have that radius, r_1, for which the pipe impedance per unit length is one quarter the column impedance. Since the latter is given by $\mu/\pi\ r_0^2 s^2$, while the pipe impedance is given by $8\mu/\pi\ r_1^4$, for the above condition we have:

$$r_1 = \sqrt{\sqrt{32}\ r_0 s} \tag{12}$$

The frit portion calculated for the immediate proximity of the column wall is also unrealistic. It shows the frit approaching the wall at a distance $t_0 = 2\sqrt{3}s$, which is the exact thickness of free space in which the impedance per unit area would equal the frit impedance. However, even if the frit were to go all the way to the wall there would be a virtually free layer of appreciably greater thickness due to the free passages around the frit particles being much larger than in the frit bulk, as is the case for the column packing itself, where a higher speed layer causes a concentration front distortion which is responsible for a large increase in the basic HETP of the packing.

The elimination of this highly damaging high speed layer at the column inner wall is believed to be one of the most important tasks of column designers, and if and when progress is made in this direction, the adoption of frit designs along the lines discussed here will prove useful.