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Grob K, Grob G & Grob K. Deactivation of glass capillary columns by silylation. Part 1: principles and basic technique. *J. High Resol. Chrom. Chrom. Commun.* 2:31-5, 1979.
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A method was described for preparing gas chromatographic glass capillary columns coated with silicone stationary phases. The most demanding step concerns pretreatment of the internal capillary wall in order to obtain thorough deactivation. The described procedure involves intensive leaching of the glass surface (20 percent HCl, 105 °C overnight), rinsing, dehydration, and silylation at very high temperature (400 °C). fine SCI® indicates that this paper has been cited in more than 155 publications, making it the most-cited article published in this journal.]

Preparation of Capillary Columns for Gas Chromatography

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The promoter for the work and the author of the paper was my father, Kurt Grob, who died in 1987. Gertrud Grob, my mother, carried out some of the column preparation work and most of the column testing. The results of the paper are based on the preparation of more than 200 capillary columns. Our laboratory had the peculiar status of a "permanent guest scientist laboratory," which primarily meant absolute freedom. For all three of us, the work rather resembled a hobby, largely carried out at odd working hours. My father was a passionate teacher of elementary chemistry for those 16 to 19 years old; my mother, previously a teacher in sewing and knitting, also worked just part-time. I worked for my thesis in a different field and had a part-time job at the Kantonales Labor. Nevertheless, my father and I produced columns at an average rate of three to four per working day (whereas the preparation of a column dragged on for about four days). My mother tested them, "nursed" the "sick" ones, and also provided a decent "funeral" for most of them.

My father experimented with silylation of glass capillary columns in the late 1960s[1]—without any success. Procedures Introducing specific functional groups, as described by others,[2] appeared to be no better. Therefore we concentrated for a long time on "inorganic" surface pretreatments, involving basic and acidic etching of glasses or deposition of selected salts. Such treatments promised to be highly thermostable (a severe problem with all "organic treatments" tested). There was also the idea that the support surface should be "hard," i.e., not allowing any penetration of solute material into the support. For these reasons, I was skeptical about returning to silylation procedures in 1977. In fact, after an initial success, producing a column of a quality never seen before, more than 50 columns were terrible, without any sign of hope. I remember joking in those days about "another day of awful columns?" Of course, there was some competition between father and son, with a score not far from 1:1; but, this time I clearly lost.

In 1976, T. Welsch[3] reported excellent deactivation of glass capillaries, applying silylation at 300 C. This temperature was generally considered crazy, but the high temperature was one of the two prerequisites for the final success (it was finally increased to 400 °C). The other key point concerned leaching of the glass surface: Only intensive treatment with acid produced the silanol groups that allowed efficient silylation. The final success was spectacular: Inertness was far better than anything produced before, and the upper temperature limit was increased from some 250 °C to beyond 350 °C, opening the way for procedures such as gas chromatographic (GC) analysis of triglycerides and to what is today called "high temperature GC."

The method was published including all the little tricks,[4] and it made a black art into something accessible to all. In fact, the method was rapidly adopted by most who prepared glass capillary columns and, with some modifications (e.g., by Blum[5]), it is still the standard procedure today. However, some disappointment followed rather rapidly. In 1979, the fused silica capillaries were introduced and rapidly created a big market. Knowledge about column preparation became a commercial issue, and just a few are left who know how to make capillary columns.

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4. **Grob K.** *Making and manipulating capillary columns for GC.* Heidelberg, Germany: Hüthig, 1986.
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