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Note

Inner surface deterioration in glass-lined tubing

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Glass-lined tubing (GLT) has become widely used in gas chromatography for injectors, capillary connectors, and various manifolds since its introduction in the

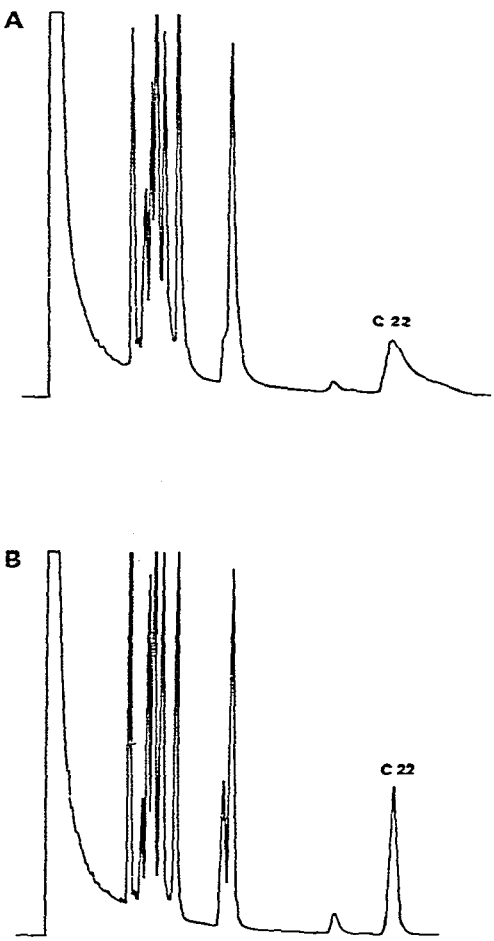

Fig. 1. Separation of monosaccharides and related compounds (as trimethylsilyl derivatives) on a 60 m × 0.5 mm I.D. glass SCOT SE-30 capillary column; temperature, 208°; helium flow, 7 ml/min. (A) Using the original injector; (B) using the same injector but with a replacement 0.7 mm I.D. inner GLT core. C_{22} indicates the *n*-alkane internal standard; mass injected, 150 ng in 0.3 μl.

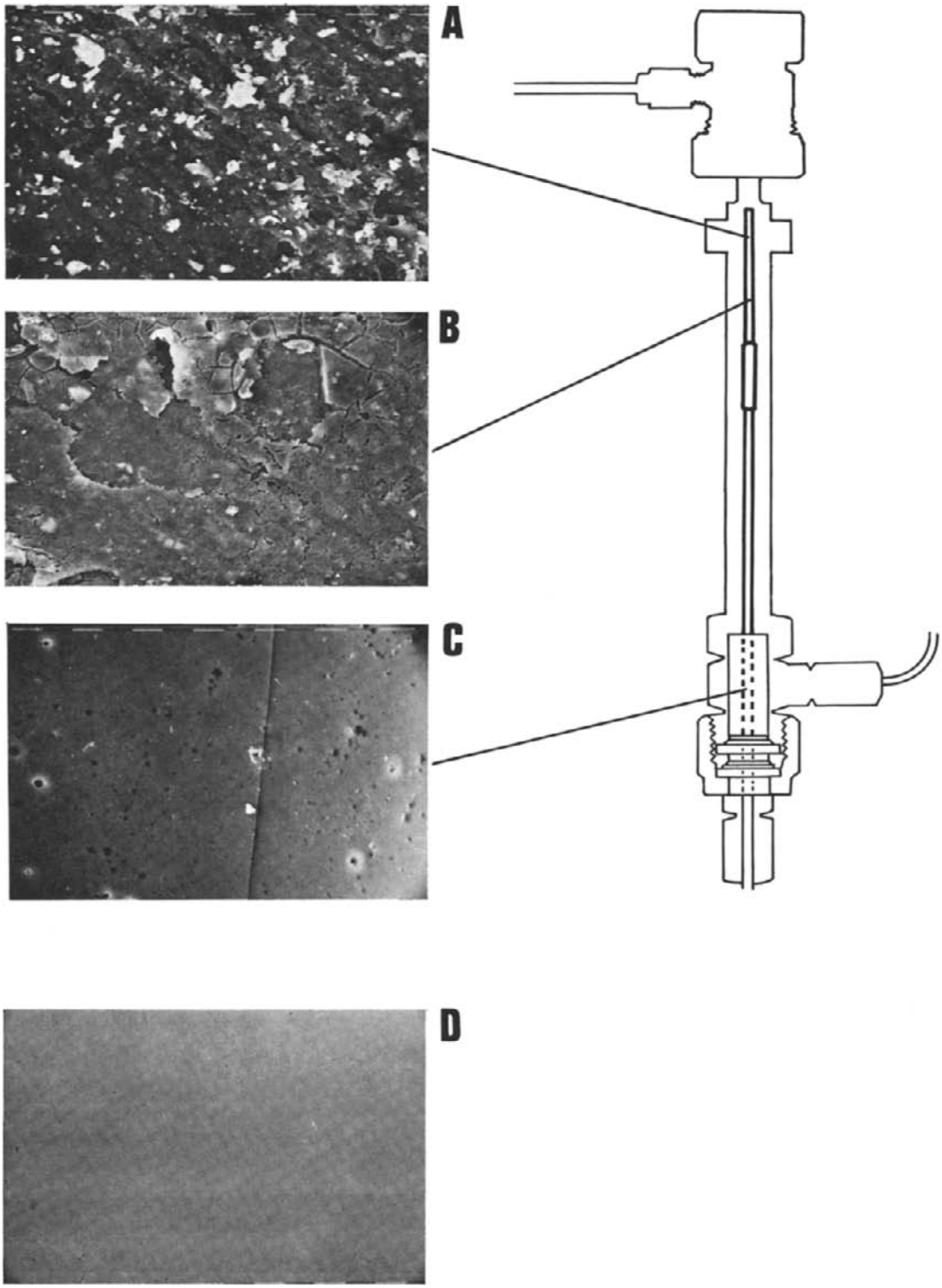

Fig. 2. Diagrammatic representation of the injector unit with the affected inner GLT core indicated in heavier outline. Arrows from each photograph indicate the point within the GLT at which the photographs were taken. Photograph D is of the inner surface of a new section of GLT for comparison. Magnifications: (A) ×900; (B) ×900; (C) ×1000; (D) ×1200. Each section of the bar scale on the photographs represents 10 μm.

early 1970s. Over the past four years we have been using a commercially available splitless injection unit with a glass support-coated open tubular (SCOT) capillary column assembly (Scientific Glass Engineering, North Melbourne, Australia) together with automatic sample injection. Lately persistent loss of overall column performance occurred, which forced us to seek the cause of this loss.

The severity of the problem is illustrated in Fig. 1A which shows both the loss of resolution with a mixture of carbohydrate derivatives and the very pronounced tailing of the *n*-C_{22} alkane used as internal standard. All parts of the chromatographic system were checked until it was finally determined that the cause lay within the injector. Involatile deposits or fractures of the glass layer were thought to be responsible. The manufacturers do specify a method for detecting cracks within the glass lining[1], but the method is hardly suitable for routine use on such assemblies. No lasting improvement was gained by various cleaning or injection techniques until finally the inner GLT core within the injector was replaced. The improvement in resolution and column efficiency is illustrated in Fig. 1B. Despite the fact that the replacement GLT used was 0.7 mm I.D. throughout (instead of 0.5 mm) column efficiency rose from N_{Eff} 14,400 to N_{Eff} 26,700 (for the C_{22} peak) under our routine operating conditions (SE-30 column, 208°, helium flow of 7.0 ml/min).

The original GLT injector core was cut open with a diamond abrasive disc for inspection and the sections sequentially cleaned by sonication in methanol, water, methanol and carbon tetrachloride to remove organic and particulate deposits. The glass lining was then examined with a scanning electron microscope at various points along its length after surface coating with gold in the normal way (Fig. 2). There is deep scarring and disruption of the surface in the region to which the needle tip penetrated, as well as deposition of irregular particles, possibly from displaced glass wall or septum or inorganic sample residues (Fig. 2A). Slightly lower down (Fig. 2B) there is further degradation of glass wall but of a different form. Here it seems as if there has been thermal shock, giving irregular "crazing", as well as regions where further chemical "etching" has removed more of the lining. Further "etching" is evident even near the base of the injector (Fig. 2C) where there is distinct pitting of the glass surface. This particular portion also includes what seems to be a linear fault in the deposition of the glass layer. The final photograph (Fig. 2D) is of the inner face of a new portion of GLT which shows a uniformly smooth surface.

It is obvious that regular checking and replacement of GLT will be essential in certain situations. It is difficult to forecast how soon performance may become unacceptable since our injection head has been in use over a four-year period, for both manual and automated injection. Sample throughput of fairly complex mixtures over that period (mainly as trimethylsilyl derivatives) is estimated at more than 20,000. It appears in retrospect that for continued good results the injector should have been refurbished much earlier or after a smaller number of samples.

ACKNOWLEDGEMENT

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REFERENCE

1 *Data sheet Reference GLT 4/75*, Scientific Glass Engineering Pty., North Melbourne, 1975.