

CHROM. 14,720

Note

Compensation of baseline drift in temperature-programmed capillary gas chromatography with electron-capture detection

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(Received December 15th, 1981)

Reduction in the standing current of an electron-capture detector due to bleeding from the stationary phase is one of the major problems in temperature programmed gas chromatography–electron-capture detection (GC–ECD)¹. The reduction may arise from trace contaminant vapours in the carrier gas or pyrolysis products of the stationary phase. Bleeding increases exponentially with temperature², producing a dramatically rising baseline at higher oven temperatures, which can make quantitation of peaks difficult. Use of non-polar phases reduces bleeding effects, while high-molecular-weight compounds with large electron affinities may cause severe baseline drift with polar phases.

The conventional approach to compensate for baseline drift, dual column analysis, is usually not practised in capillary GC–ECD owing to the high cost of duplicating the detector and column. Recently, a single column compensation method, which stores the column bleed profile during a blank run, was presented³.

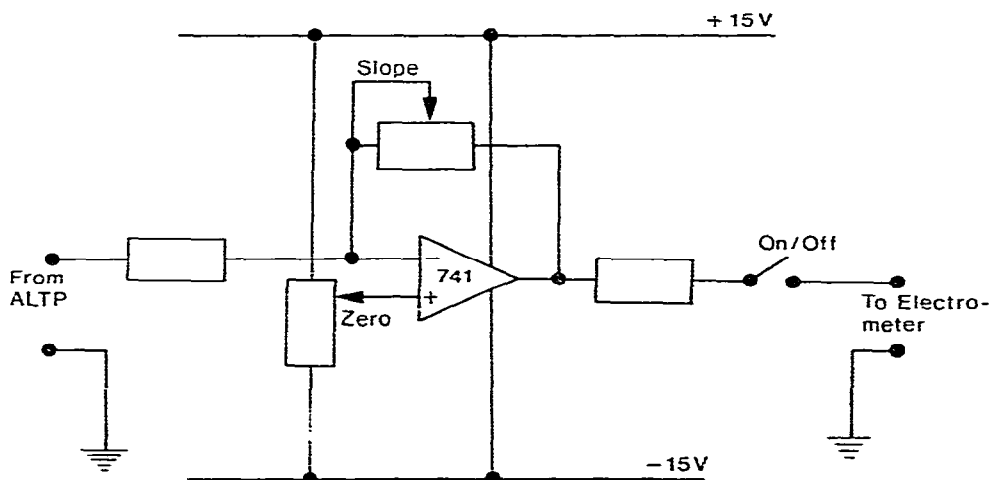


Fig. 1. Wiring diagram for a linear compensation of baseline drift.

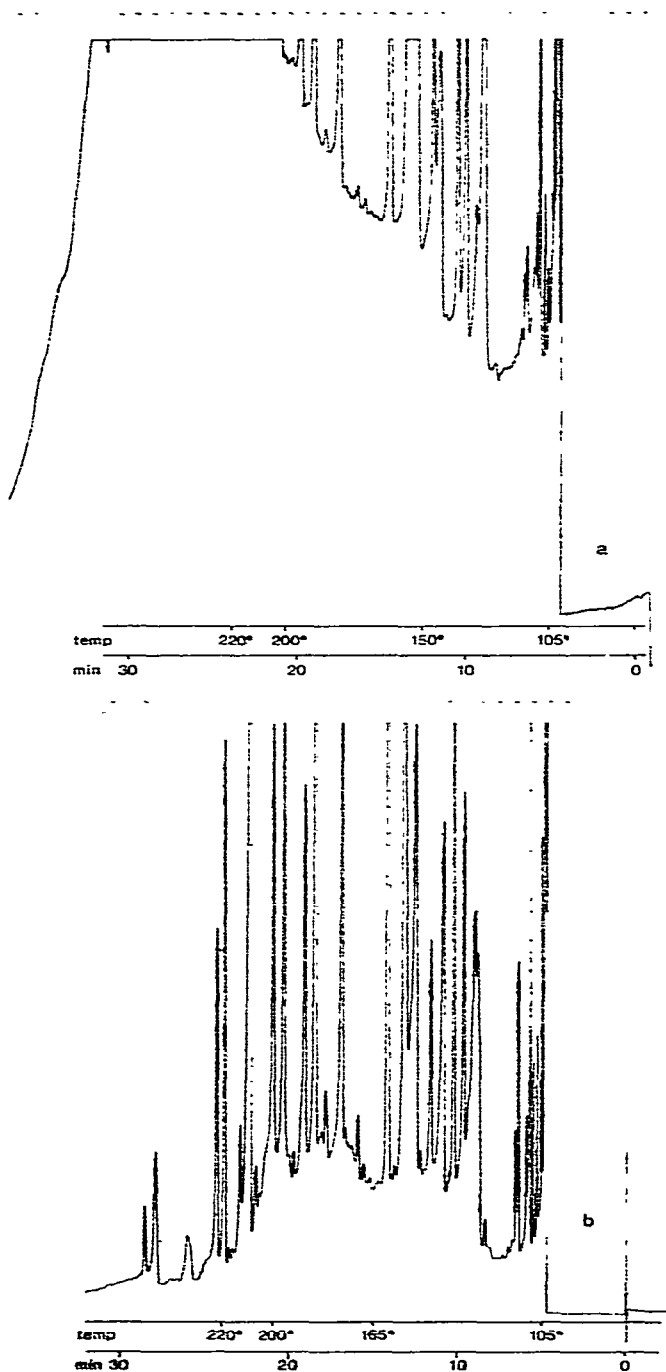


Fig. 2. Gas chromatograms after injection of a 0.5- μ l sample of amino acids on a 50-m SCOT column with temperature program and a ^{63}Ni detector; without (a) and with (b) baseline correction.

We here report on a simple displacement of the electrometer voltage to compensate for baseline drift in capillary GC-ECD.

The experiments were performed with a Varian 3700 gas chromatograph equipped with a ^{63}Ni detector. Baseline compensation was achieved by utilizing a voltage in the automatic linear temperature programmer (ALTP) (Fig. 1). The voltage, which decreases proportionally with increasing temperature, was inverted and amplified so that it cancelled out the drift caused by temperature programming when fed into the electrometer. The slope control sets the gain for the correct amount of compensation and the zero control is for zeroing at the initial temperature. With the On/Off switch the circuit can be switched out when no compensation is required.

The performance of the compensation mechanism was demonstrated with a sample of amino acids, a class of compounds with a wide range of boiling points and which consequently require temperature-programmed separation. Owing to the low volatility of amino acids, suitable derivatives have to be prepared for gas chromatography. After purification by ion-exchange, the amino acids were converted into their corresponding heptafluoroisobutyl derivatives⁴. These are highly electron absorbing and volatile and have the potential of analysing amino acids at the trace levels found in biological microenvironments⁵.

Derivatives were separated on an SE30/OV17 SCOT column (50 m \times 0.5 mm I.D.). The oven was maintained at 105°C for 5 min, then programmed at 6°C/min to 220°C and held there for 10 min. Injections were made with no splitting at 250°C. Nitrogen was used as a carrier gas at 4.5 ml/min and the ECD was maintained at 300°C. The volume injected was 0.5 μl , representing *ca.* 0.76 pmole for the large peaks.

At the attenuation and range used ($16 \cdot 10^{-10}$ A/mV) the uncompensated analysis shows a heavy bleed and the baseline runs off the scale (Fig. 2a), while the compensation keeps the baseline in its starting position (Fig. 2b), thus facilitating integration of the peaks.

The compensation method was successfully applied to samples with less than 10 fmole of substance, but at even lower levels a baseline hump between 110 and 150°C could not be avoided if the last eluted peaks were not to be overcompensated for and thus appear below the baseline. This effect was certainly due to the exponential relationship between bleeding and temperature. For these cases an exponentially compensating voltage may be applied to the electrometer.

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