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Note

Gas chromatographic determination of methanal traces in presence of other volatile carbonyl compounds

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The determination of methanal in products such as flavourants containing large amounts of volatile carbonyl compounds is, in most cases, impossible with the classical optical methods using reagents such as acetylacetone, chromotropic acid, albumin–iron reagent, 4-amino-3-hydrazino-5-mercapto, 1,2,4-triazol, etc. The direct gas-chromatographic detection on molecular sieve is not sufficiently sensitive. For these reasons, we have adapted the gas-chromatographic separation of 2,4-dinitrophenylhydrazone suggested by Soukup *et al.*[1] to obtain a method allowing trace determination at the 10 ppm level.

EXPERIMENTAL

Reagents

The reagents used were as follows: 2,4-dinitrophenylhydrazine (DNP); sulphuric acid; DNP reagent: dissolve 1.0 g DNP in 20 ml sulphuric acid and a few drops of water, and dilute slowly to 100 ml with water; benzene; all were of analytical grade.

Apparatus

The apparatus consisted of: test tubes (3 ml); electric stirrer for test tubes (Vortex-Genie); 100 and 600 μl piston pipettes (Eppendorf) gas chromatograph (Perkin-Elmer 900 or similar); glass column (4 m × $\frac{1}{8}$ in. I.D.); stationary phase: 4% SE-30 + 4% OV-17 on Chromosorb W HP (100–120 mesh) or 10% OV-101 on Chromosorb W HP (100–120 mesh). The operating temperature was programmed from 150–220° (6°/min) with the injector and manifold at 300°. The carrier gas was nitrogen (30 ml/min) and detection was by flame ionisation.

METHOD

In a test tube, add 600 μl of DNP reagent to 100 μl of sample. Mix well and allow to stand for 15 h (temperature: 1–3°). In the same test tube, just before the injection, extract the derivative in 100 μl benzene. Ensure an excess of reagent has

been used: the water phase remains yellow after the extraction. Inject 2 μl of benzene solution.

For quantitative determination, use internal standards of methanal.

RESULTS

This method was found suitable for determination of methanal at the 10 ppm level in concentrated liquid apple and smoke flavours.

REFERENCE

1 R. J. Soukup, R. J. Scarpellino and E. Danielczik, *Anal. Chem.*, 36 (1964) 2255.