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Note

A simple method for rapid alkali flame ionisation detector optimisation

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A major disadvantage of alkali flame ionisation detectors (AFIDs) is that sensitivity is critically dependent upon a number of parameters, in particular, the alkali salt–flame separation distance[1]. The practical operation of an AFID requires in many cases, that the salt–flame separation is optimised daily; usually by a tedious, time-consuming sequence of alternate sample injections and adjustment of a detector parameter. This note reports a simple method for rapid AFID optimisation (AFIDO).

The principle of the method is to introduce a slowly varying amount of sample material to an AFID and to adjust continuously a detector parameter, such as: alkali salt position[1], polarising voltage[2] or flame gas flow-rates[3], until a maximum response is obtained. For convenience, sample material is introduced using a hypodermic syringe needle, inserted into the injection port in the usual manner.

EXPERIMENTAL

A Pye Unicam GCV chromatograph, fitted with a GCV nitrogen AFID and a 1.5-m 10% E30 glass column were set up according to the manufacturer's instructions. The nitrogen detector was of the three-electrode (RbCl) type. The flame was set with the following gas flow ratios: hydrogen–nitrogen–air (30:30:300). The AFIDO apparatus was constructed by filling a 7-cm, 100-μl syringe needle with 2,6-dinitrotoluene.

Chromatographic conditions were as follows: detector temperature, 200°; column temperature, 170°; injector temperature, 100°.

The AFIDO was initiated by inserting the filled syringe needle to a depth of 5 cm into the injection port of the chromatograph. This allowed 2,6-dinitrotoluene to "bleed" slowly onto the column. Penetration of the column packing material by the needle, was found not to affect the optimisation.

After a period of 10 min, during which time the flow of 2,6-dinitrotoluene to the nitrogen detector was established, the position of the alkali salt was carefully adjusted up and down, by means of an external screw, until a maximum response was obtained. The time taken for this stage of the optimisation was about 20 sec. The AFIDO needle was then removed, leaving the nitrogen detector in a fully optimised condition, as shown by analysing a mixture of pyridine, *p*-nitrotoluene and *n*-hexylamine. Each compound gave the maximum response.

CONCLUSIONS

The AFIDO is a simple, rapid method of general applicability, for obtaining the most favourable response from an AFID. By a suitable choice of sample material, this method of optimisation may be extended to other chromatographic detector systems, such as the flame ionisation detector and the flame photometric detector, which may require optimisation at regular intervals to ensure maximum sensitivity.

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