

Characteristics of Flame Ionization Detection for the Quantitative Analysis of Complex Organic Mixtures

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INTRODUCTION

A flame ionization detector (FID) is the most commonly used detector for gas chromatography due to its high sensitivity for organic compounds, wide linear dynamic range, and almost zero dead volume. The FID responses of homologues are proportional to the number of carbon atoms or molecular weight. The relative weight response factors are very similar for a variety of hydrocarbons regardless of compound type or molecular weight. Thus, the weight percentage can easily be calculated from the area percentage. However, FID still has its own limitations. If the hydrogen atoms in hydrocarbons are substituted by some functional groups or heteroatoms, the FID responses of the substituted ones will decrease. That is, the FID responses are very dependent upon compound structures and the presence of heteroatoms (1). As a result, in quantitative determination of different components, it is necessary to calibrate them with the known compounds. In this paper, the FID relative weight response factors of hydrocarbons and those of organic compounds containing heteroatoms, such as oxygen, chlorine, and bromine, have been determined. Furthermore, when the FID relative weight response factors obtained by the authors and by Tong et al. (2) are converted into relative carbon weight response factors, surprisingly good carbon-regularity resulted.

EXPERIMENTAL SECTION

Determinations of the FID responses of standard compounds were carried on a 1091C-C gas chromatograph equipped with a FID (Shanghai Analytical Instruments Works, Shanghai, People's Republic of China). A 30 m $\times$ 0.32 mm i.d., 0.25- μ m film thickness DB-5 fused silica capillary column and a 30 m $\times$ 0.315 i.d., 0.25- μ m film thickness SE-52 fused silica capillary column (J&W Scientific, Inc.) were used. FID signals (peak areas) of compounds were recorded by 3390A integrator (Hewlett-Packard).

The FID relative response factor and relative carbon response factor of a compound are calculated as follows:

$$\text{FID relative response factor} = \frac{\text{FID response factor of compound}}{\text{FID response factor of reference}} \quad (1)$$

where

$$\text{FID response factor} = \frac{\text{peak area of compound}}{\text{quantity of compound injected}} \quad (2)$$

and

$$\text{FID relative carbon response factor} = \frac{\text{FID carbon response factor of compound}}{\text{FID carbon response factor of reference}} \quad (3)$$

where

$$\begin{aligned} \text{FID carbon response factor of compound} &= \\ & \frac{\text{peak area of compound} / [\text{quantity of compound injected} \times \\ & \quad (\text{total C at wt in compd} / \text{mol wt})]}{\text{molecular weight}} \\ \text{FID response factor} &\propto \frac{\text{total C at wt in compd}}{\text{molecular weight}} \quad (4) \end{aligned}$$

RESULTS AND DISCUSSION

The FID relative weight response factors and FID relative carbon weight response factors for a variety of hydrocarbons, chlorohydrocarbons, bromohydrocarbons, and oxygenated hydrocarbons are tabulated in Table I. Table I shows that compounds with various structures have very different FID relative weight response factors and the quantitative determination is rather laborious because a number of calibration factors must be introduced. However, when FID relative responses are converted into relative carbon responses, a good linear regularity is found. The FID relative carbon response factors are negligibly affected by the presence of heteroatoms.

When the results of FID response factors obtained by Tong and Karasek (2) are converted into FID carbon response factors (Table I), good carbon regularity is also found. The FID relative carbon responses of dimethyl and diethyl phthalates (Table II) are much smaller than those of others due to the higher percentage of oxygen atoms in the two compounds.

One of the possible mechanisms in the flame ionization detection scheme suggests that every hydrocarbon is degraded to the same distribution of single-carbon radicals before ionization takes place. The response of the FID to an organic compound is due to the generation of ion via the chemical ionization process when an organic sample containing $-\text{CH}_x$ groups is introduced into the flame (3).



This flame process generates ions in proportion to the number of oxidizable carbon atoms in the eluting compound. For compounds substituted by halogen atoms, it is supposed that when halogen atoms leave the compound, the decomposition of the remainder of the compound should be the same as that of the pure hydrocarbons. That is



That the strength of chemical bond C-H (X is chlorine or bromine) is weaker than that of the C-C bond (4) also supports the above proposition, which has explained why the compounds containing chlorine or bromine atoms have good FID carbon regularity. For this reason, a new calculation method for FID quantitative analysis is recommended, i.e.

$$W_i\% = \frac{A_i M_i / C_i}{\sum A_i M_i / C_i} \times 100 \quad (8)$$

where W_i is the weight percent of the compound determined, A_i is the peak area of the compound determined, M_i is the molecular weight of the compound determined, and C_i is the total atomic weight of carbon in the compound determined.

The concept of the effective carbon number (ECN) was introduced many years ago (1) to explain the observed flame ionization responses obtained from analyzing the isomeric or homologous series of organic compounds. It provides a way to calculate relative response factors for compounds whose

Quantitative Analysis Of Organic Mixtures Part 1

General Principles

L Cohen

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