

RJR INTER-OFFICE MEMORANDUM

TO: Dr. Brian Lawrence

FROM: Ken Shu

DATE: March 30, 1988

RE: QUANTITATIVE DETERMINATION OF MENTHOL AND CARVONE LEVELS IN IFF SPEARMINT FLAVOR (TOOTHPASTE FLAVOR OIL)

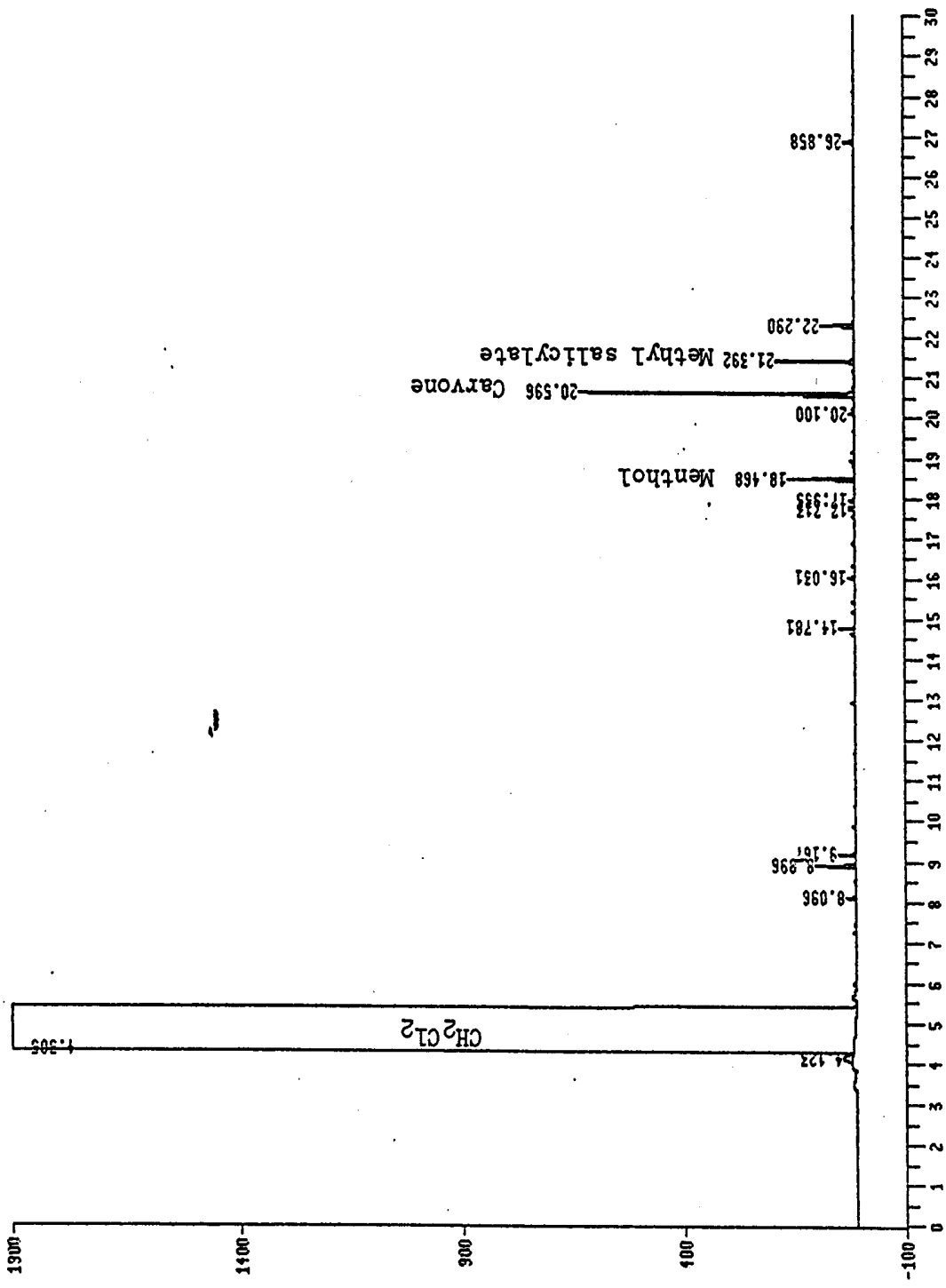
In order to correlate the TF aging study data with the total spearmint flavor amount in the pellet/filter section, the accurate menthol and carvone levels in spearmint flavor have been determined to be 10.03% and 49.53% respectively by the internal standard method on a DBWAX fused silica column (60 m x 0.32 mm, 50°C to 188°C at 6°C/min) using a GC/autosampler instrument.

Spearmint flavor (5 mg-25 mg) was diluted in CH₂Cl₂ prior to the analysis. Methyl salicylate was used as the internal standard chemical. Figure 1 shows the typical gas chromatogram of the spearmint flavor in CH₂CH₂ indicating menthol, carvone and methyl salicylate peaks, Figure 2 the calibration curves for menthol and carvone. Table 1 is the summary of the quantitative results.

It is noteworthy that the data obtained from this analysis by the internal standard method (menthol: carvone about 1:5) are different from those obtained from the previous analysis (reported on February 23, 1988) by area percent method (menthol: carvone about 1:3). Apparently, the internal standard method is more accurate because the response factors for menthol and carvone~~s~~ have been calibrated during the analysis.

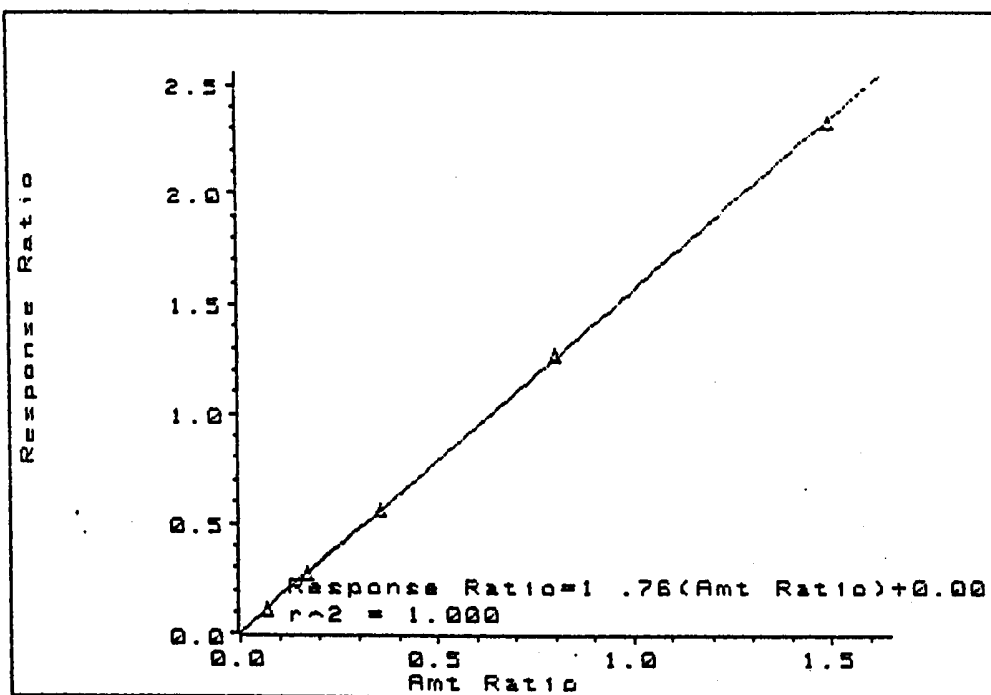
Ken Shu

FIGURE 1 TYPICAL GC OF THE SPEARMINT FLAVOR IN CH₂CL₂

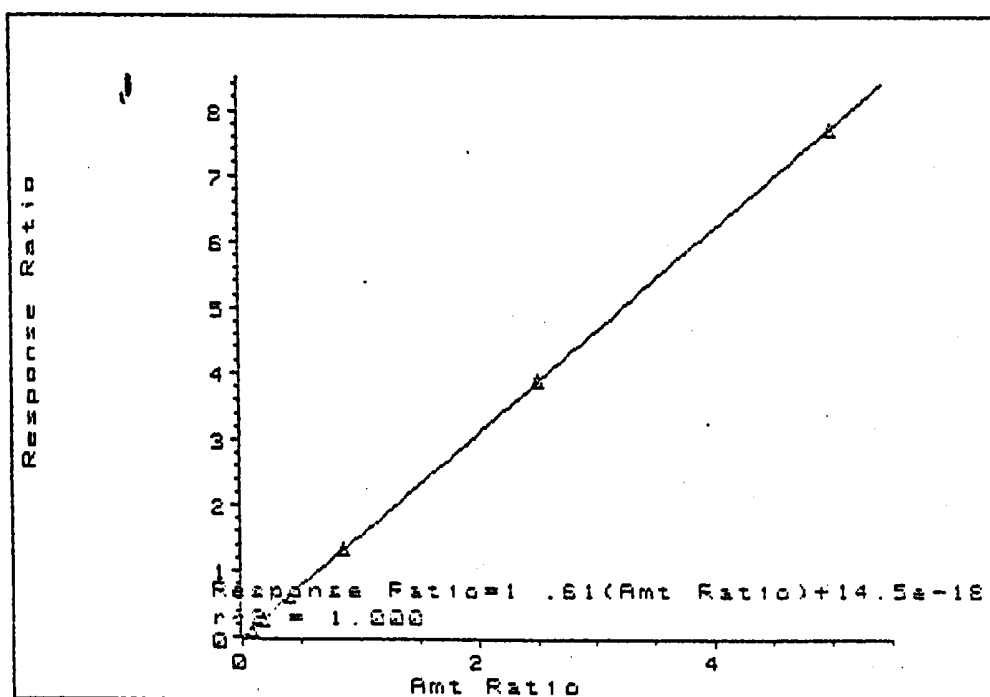


1: Sig. 2 of DATA:0324412A.D

End of plot. Time = 0.00 to 30.00 minutes Chart speed = 0.66 cm/min



A



B

FIGURE 2 CALIBRATION CURVES FOR MENTHOL(A) & CARVONE(B)

Table 1. Menthol and Carvone Levels Quantitized from the Spearmint Flavor

| Sample # | Sample amount (mg) | Menthol | | Carvone | | Total % in sample |
|----------|-----------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|----------------------|
| | | Amount, % in sample (mg) | Amount, % in sample (mg) | Amount, % in sample (mg) | Amount, % in sample (mg) | |
| 1 | 5 | 0.495 | 9.908 | 2.504 | 50.085 | 59.993 |
| | | 0.496 | 9.914 | 2.486 | 49.713 | 59.627 |
| 2 | 10 | 1.009 | 10.090 | 4.976 | 49.758 | 59.848 |
| | | 1.007 | 10.076 | 4.973 | 49.728 | 59.804 |
| 3 | 25 | 2.530 | 10.120 | 12.257 | 49.029 | 59.149 |
| | | 2.523 | 10.091 | 12.215 | 48.858 | 58.949 |
| Ave. | | | 10.03% | | 49.53% | 59.96% |