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# Optimizing Sensitivity of SIM Mode of GC/MS Analysis for EPA's TO-14 Air Toxics Method

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Guidelines for determining volatile organic compounds (VOCs) at ambient levels in air are published in Method TO-14 of the U.S. Environmental Protection Agency's "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air." To achieve the sensitivity required for sub-part-per-billion-by-volume (ppbv) determinations of VOCs, TO-14 recommends selective ion monitoring (SIM) mode of operation when a conventional quadrupole mass spectrometer is used as the analytical finish. This paper describes the implementation of SIM mode of analysis and presents the rationale and methods for optimizing SIM parameters for maximum sensitivity when analyzing whole-air samples for trace levels of targeted VOCs.

The determination of a specific set of nonpolar volatile organic compounds (VOCs) at ambient, trace levels has been described in the EPA guidance document "Compendium of Methods

for the Determination of Toxic Organic Compounds in Ambient Air, Method TO-14."<sup>1</sup> The recommended analytical strategy uses cryogenic preconcentration of analytes with subsequent gas chromatographic (GC) separation and mass spectrometric (MS) detection. Because the amounts of target compounds found in ambient air are generally small (typically less than 5 parts per billion by volume, ppbv), this methodology requires detecting nanogram and subnanogram quantities. With existing quadrupole mass spectrometers using electron impact ionization, full-scan spectral detection (typically to 350 amu) does not have the sensitivity required for ambient air measurements. To address the sensitivity issue, Method TO-14 recommends the use of a selective ion monitoring (SIM) spectrometric technique. This method programs the mass spectrometer to detect only certain ionic fragments at certain retention times for the target compounds and to disregard all others. Typically, two or three of the major ions of each compound are chosen. This increases the signal-to-noise ratio (S/N) of compound detection to allow a lower limit of detection of 0.10 ppbv for all 40 target analytes on the current TO-14 list (see Table I). The trade-off for this increased sensitivity is a detection capability that is restricted to the targeted compounds; all other compounds are not detected unless they exhibit a coincidental (uncommon) overlap in ion fragment and chromatographic elution time. Recent developments in the Superfund and the Resource Conservation and Recov-

ery Act (RCRA) programs, as well as the development of a  $10^{-6}$  risk level (defined as a concentration that presents a one in a million cancer risk for lifetime exposure) for some specific air toxics, require that new compounds be added to the TO-14 list and that the method detection limits be lowered. The optimization of sensitivity and specificity of the SIM-type TO-14 analysis are discussed below.

## Experimental Section

### Discussion of MS Parameters

The existing TO-14 method allows for 40 specific VOCs, each detected by two or three ions, as shown in Table I (in some applications, the third ion per compound is not used). Typically, ions are detected in groups of 6 to 12 in any given portion of the chromatogram. The mass spectrometer is programmed to automatically begin a search for the next group of ions within an elution time window that allows for minor retention time shifts, typically with a safety factor of at least 0.2 min. The specific ions and their respective groups that were used in our laboratory are given in Table I. Chromatographic differences among laboratories may result in different elution times; however, if similar temperatures and GC columns are used, the order of elution should remain the same.

Three adjustable sets of acquisition conditions exist in the separation and detection of the analytes: (1) chromatographic separation in time, (2) number of ions being searched for during a given time interval, and (3) the mass spectrometer acquisition (dwell) time, which is the time spent accumulating data for an individual ion. These parameters are interrelated, and all affect the sensitivity of the analysis:

1. The first parameter set is the least adjustable for this application; separation is determined by the

### Implications

EPA's Method TO-14 is becoming the accepted method for determining trace-level, non-polar, volatile organic compounds (VOCs) in ambient air for indoor air, ambient air, and other regional and state programs. In addition, the basic method is in revision to become part of the Statement of Work for the Superfund Contract Laboratory Program and also will be used to address some of the provisions in the 1991 Clean Air Act Amendment legislation. As such, many laboratories are adding the TO-14 technology to their analytical capability. This paper presents the rationale and general procedure for optimizing the sensitivity of mass spectrometric detection specifically for target compound determination via selective ion monitoring (SIM). Because SIM is specified as the preferred analytical method, yet the topic of SIM optimization is not included in the TO-14 methodology, this paper provides guidance for implementing optimized analytical performance for determining VOCs.



**Table I.** Target compounds, retention times, and SIM ions for U.S. EPA Method TO-14 as implemented in AREAL, U.S. EPA, Research Triangle Park, NC.

Group	Compound	Retention Time (minutes)	SIM Ions
1	dichlorodifluoromethane	5.01	85, 87
	methyl chloride	5.69	50, 52
	1,2-dichloro-1,1,2,2-tetrafluoroethane	6.55	85, 135, 87
	vinyl chloride	6.71	62, 27, 64
2	methyl bromide	7.83	94, 96
	ethyl chloride	8.43	64, 29, 27
	trichlorofluoromethane	9.97	101, 103
3	1,1-dichloroethene	10.93	61, 96, 63
	dichloromethane	11.21	49, 84, 86
	1,1,2-trichloro-1,2,2-trifluoroethane	11.60	151, 101, 103
4	1,1-dichloroethane	12.50	63, 27, 65
	cis-1,2-dichloroethene	13.40	61, 96, 98
	trichloromethane	13.75	83, 85, 47
	1,2-dichloroethane	14.39	62, 27, 64
5	1,1,1-trichloroethane	14.62	97, 99, 61
	benzene	15.04	78, 77, 50
	carbon tetrachloride	15.18	117, 119
	1,2-dichloropropane	15.83	63, 41, 62
6	trichloroethene	16.10	130, 132, 95
	cis-1,3-dichloropropene	16.96	75, 39, 77
	trans-1,3-dichloropropene	17.49	75, 39, 77
	1,1,2-trichloroethane	17.61	97, 83, 61
7	toluene	17.86	91, 92
	1,2-dibromoethane	18.48	107, 109, 27
	tetrachloroethene	19.01	166, 164, 131
	chlorobenzene	19.73	112, 77, 114
	ethyl benzene	20.20	91, 106
	m,p-xylene	20.41	91, 106
	styrene	20.81	104, 78, 103
	1,1,2,2-tetrachloroethane	20.92	83, 85
	o-xylene	20.92	91, 106
	4-ethyl toluene	22.53	105, 120
8	1,3,5-trimethylbenzene	22.65	105, 120
	1,2,4-trimethylbenzene	23.18	105, 120
	m-dichlorobenzene	23.31	146, 148, 111
	benzyl chloride	23.32	91, 126
9	p-dichlorobenzene	23.41	146, 148, 111
	o-dichlorobenzene	23.88	146, 148, 111
	1,2,4-trichlorobenzene	26.71	180, 182, 184
	hexachlorobutadiene	27.68	225, 227, 223

GC temperature program, column dimension, carrier gas flow rate, and column chemistry. For these analytes, column chemistry is restricted to the nonpolar stationary phases so that only the temperature program, flow rate, and the column dimensions can be adjusted. Separation can be improved by decreasing the rate of the temperature program, increasing column flow, and using longer capillary columns, all of which have the negative trade-offs of increased analysis time and peak shape degradation. These trade-offs can be optimized by using 50-m  $\times$  0.32-mm-i.d. or 30-m  $\times$  0.53-mm-i.d. methyl silicon capillary columns coupled to a -50 to +200°C temperature ramp at 8°C/min at 2 to 4 cm<sup>3</sup>/min, as given

in TO-14.

- The number of ions being searched for in any given analysis time interval helps determine the "duty cycle," or the percentage of the time any individual ion is sought. Though the duty cycle is not an exactly (inverse) linear function with respect to the ion number, reducing the number of ions being searched for increases the duty cycle for each individual ion and therefore increases the sensitivity for that ion. Thus, given a fixed number of analytes and representative ions, the duty cycle can be increased by decreasing the number of ions per group used in the total GC run. As a minimum, individual ion groups of two or three ions could be used for each individual compound; this is not feasible in general because of the chromatographic coelution or close elution times of some of the analytes.
- The dwell time parameter can be set to adjust the mass spectral integration time for any individual ion before the spectrometer acquires data for the next ion. This is a subtle adjustment; its optimization is determined by (1) GC parameters and (2) the ion group sizes. The dwell time setting is analogous to the time constant adjustment of analog chart recorders. A large dwell time tends to damp out noise but also averages out the analytical signal, whereas a small dwell time follows the analytical signal accurately but also records the attendant noise. For this application, the dwell time must be adjusted to achieve an optimal sampling rate; that is, it must be maximized to

**Table II.** Data acquisition rate and efficiency as a function of ion group size, dwell time, and operating system.<sup>a</sup>

Computer System	Number of Ions	20-ms Dwell Time		200-ms Dwell Time		800-ms Dwell Time	
		Rate (Hz)	Duty (Cycle(%)) <sup>b</sup>	Rate (Hz)	Duty (Cycle(%))	Rate (Hz)	Duty (Cycle(%))
HP-UNIX	16	1.8	3.6	0.3	5.8	0.1	6.4
	8	3.5	7.1	0.6	11.8	0.2	12.8
	4	6.8	13.6	1.2	23.6	0.3	24.6
	2	12.2	24.4	2.3	46.2	0.6	49.6
	1	22.2	44.4	4.4	88.8	1.2	97.6
HP-Pascal	16	1.6	3.2	0.3	6.0	0.1	8.0
	8	2.4	4.8	0.5	10.0	0.1	8.0
	4	3.1	6.2	0.9	18.0	0.3	24.0
	2	3.7	7.4	1.6	32.0	0.5	40.0
	1	4.0	8.0	2.3	46.0	0.9	72.0

<sup>a</sup> Hz rates are rounded to the nearest tenth for UNIX to be comparable to Pascal data.

<sup>b</sup> Duty cycle is the percentage of the time spent acquiring data for an individual ion.



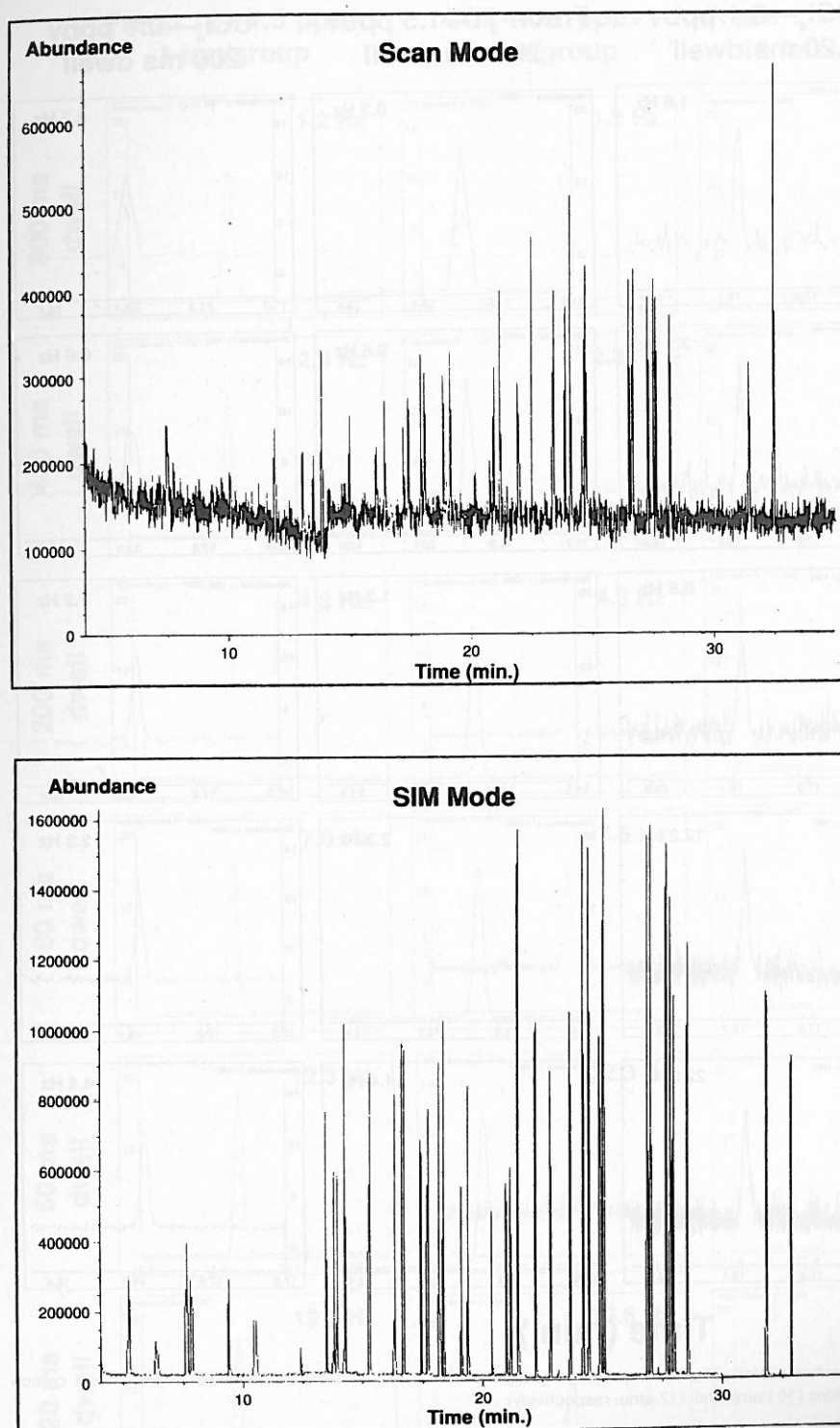


Figure 1. Comparison of scan and SIM modes as applied to a calibration sample.

smooth noise without statistically missing the apex of an eluting chromatographic peak. For the automatic peak integration programs of the mass spectrometers used for this work, a sampling rate of about 10 to 20 points per chromatographic peak is required, according to the

manufacturer.<sup>2</sup> A general discussion of mass spectrometer scan rates is available,<sup>3</sup> as is a more specific mathematical treatment of sampling rates and their relation to SIM quantitation errors.<sup>4</sup> The most applicable literature found by the authors is a discussion by Mat-

thews and Hayes,<sup>5</sup> wherein the mass cycling error is empirically determined for a variety of situations; for tailing peak shapes and a unidirectional scan, the minimum number of required points to achieve less than 0.5% average error ranges from 4 to 10. For the purposes of this paper, the theoretical target value for this parameter is 10; however, the actual sampling rate used is generally a compromise resulting from the combination of physical chromatographic parameters (peak width and shape), electronic parameters (quadrupole settling time and recycling time), duty cycle and dwell time, and computer data acquisition and storage times.

#### Optimization Tests

A number of tests were performed to determine the extent to which the TO-14 methodology could be optimized for sensitivity. Initially, calibration samples (at 10-ppbv levels) were analyzed under the scan mode and the conventional SIM mode to provide baseline information. Then, ambient air was used to prepare low-level test samples for parameter optimization. These were prepared simultaneously so that all subsequent tests were performed on a common ambient air sample. Chromatographic parameters were not modified; the column used was a 50-m  $\times$  0.32-mm-i.d. cross-linked methyl silicon capillary column; oven temperature programming was  $-65^{\circ}\text{C}$  initial setting, 2 min hold,  $8^{\circ}\text{C}/\text{min}$  ramp to  $200^{\circ}\text{C}$ . The analytical equipment consisted of a Nutech 320-1 cryogenic preconcentrator (Nutech Corp, Durham, NC) and a Hewlett-Packard (HP) GC/MS system that used an HP-5890 GC with a direct interface to an HP-5970B mass selective detector MS (Hewlett-Packard Company, Palo Alto, CA). Analytical work was performed by using the HP-9000 series 300 computer and a UNIX-based operating system for data acquisition; some comparison tests were performed with an identical GC/MS system using the Pascal 3.2 operating system running on an HP-300 series computer. The GC/MS system function details concerning sample handling and compound determination are described in the literature.<sup>6-9</sup>

The initial optimization tests investigated the effect of ion group size at fixed dwell times. Trichlorofluoromethane (Freon 11) and carbon tetrachloride ( $\text{CCl}_4$ ) were chosen as representative of the TO-14 chromatography, and their chromatographic peaks are depicted in subsequent figures. They are conveniently present in es-



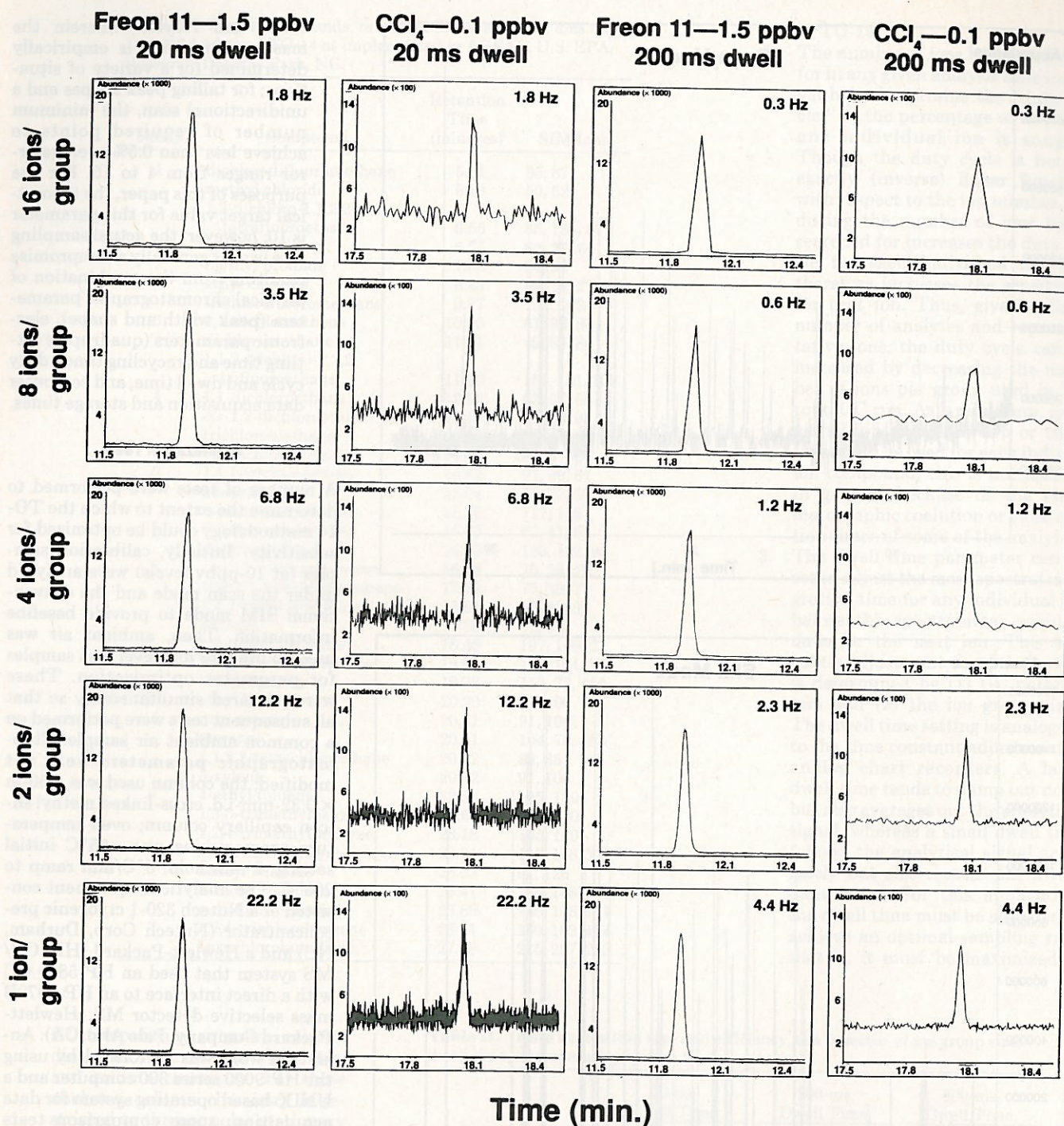


Figure 2. Representative results from SIM analyses performed for different sizes of ion groups for 20-ms and 200-ms dwell times. Freon 11 and carbon tetrachloride (CCl<sub>4</sub>) peaks are plotted for their base peak ions (101 amu and 117 amu, respectively).

essentially all ambient air samples; as such they are good examples of TO-14 performance. For the sake of brevity and clarity in the figures and subsequent text, the two compounds are referred to by their short names, Freon 11 and CCl<sub>4</sub>. A series of SIM runs was performed using identical ambient air samples in which two ion groups were used, one which included the characteristic ions of Freon 11 (101, 103) and the other which included the characteristic ions of CCl<sub>4</sub> (117, 119). Initially,

16 ions per group were acquired; then, in successive runs, the number of ions per group was reduced to 8, 4, 2, and 1. The dwell time was held constant at 20 ms per ion; this was repeated with a constant dwell time of 200 ms per ion and again at 800 ms per ion.

Next, the effect of dwell time was investigated. Again, the same samples were used, and Freon 11 and CCl<sub>4</sub> were the representative compounds. The first series of runs was performed by using 16 ions per group and dwell times

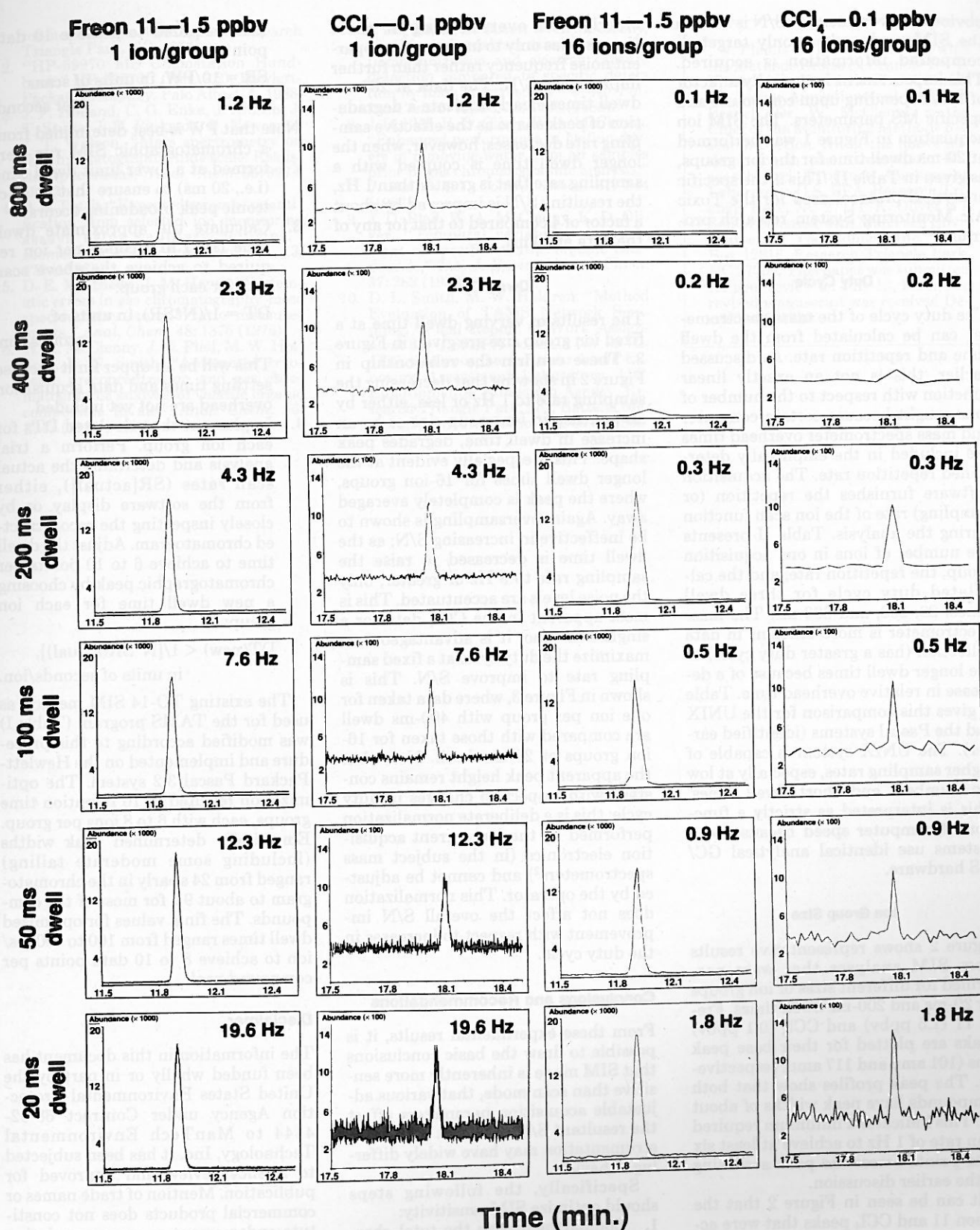
of 20, 50, 100, 200, 400, and 800 ms per ion. This series was repeated but with only one ion per group.

### Discussion of Results

#### SIM vs. Scan

The comparison of scan and SIM modes as applied to a calibration sample of the 40 target VOCs at the 10-ppbv level is presented in Figure 1. An





**Figure 3.** Representative results from SIM analyses performed for various dwell times at fixed ion group sizes of 1 and 16 ions per group. Freon 11 and carbon tetrachloride ( $\text{CCl}_4$ ) peaks are plotted for their base peak ions (101 amu and 117 amu, respectively). Sampling rate is shown for each spectrum in Hz.



obvious improvement in S/N is seen in the SIM mode, where only targeted-compound information is acquired. This improvement is typically a factor of 100, depending upon compound and specific MS parameters. The SIM ion acquisition in Figure 1 was performed at 20-ms dwell time for the ion groups, as given in Table II. This is the specific analytical protocol used for the Toxic Air Monitoring System research program.<sup>10</sup>

#### Duty Cycle

The duty cycle of the mass spectrometer can be calculated from the dwell time and repetition rate. As discussed earlier, this is not an exactly linear function with respect to the number of ions sought because certain computer and mass spectrometer overhead times are included in the empirically determined repetition rate. The acquisition software furnishes the repetition (or sampling) rate of the ion scan function during the analysis. Table II presents the number of ions in one acquisition group, the repetition rate, and the calculated duty cycle for three dwell times: 20, 200, and 800 ms. The mass spectrometer is more efficient in data collection (has a greater duty cycle) at the longer dwell times because of a decrease in relative overhead time. Table II gives this comparison for the UNIX and the Pascal systems (identified earlier). The UNIX system is capable of higher sampling rates, especially at low ion numbers and short dwell times. This is interpreted as strictly a function of computer speed because both systems use identical analytical GC/MS hardware.

#### Ion Group Size

Figure 2 shows representative results from SIM analyses that were performed for different sizes of ion groups for 20-ms and 200-ms dwell times. Freon 11 (1.5 ppbv) and CCl<sub>4</sub> (0.1 ppbv) peaks are plotted for their base peak ions (101 amu and 117 amu, respectively). The peak profiles show that both compounds have peak widths of about 6 s. This indicates a minimum required scan rate of 1 Hz to achieve at least six data points across the peak according to the earlier discussion.

It can be seen in Figure 2 that the Freon 11 and CCl<sub>4</sub> peaks that were acquired at 20-ms dwell times are not improved greatly by decreasing the number of ions per group; this is expected because even at 16 ions per group the scan rate is about 1.8 Hz, which achieves more than 10 data points per peak. The CCl<sub>4</sub> data at 20 ms show

clearly that oversampling at short times serves only to increase the apparent noise frequency rather than further improve the S/N. The data at 200-ms dwell times clearly indicate a degradation of peak shape as the effective sampling rate decreases; however, when the longer dwell time is coupled with a sampling rate that is greater than 1 Hz, the resulting S/N is improved by about a factor of 4 compared to that for any of the data at 20 ms.

#### Dwell Time

The results of varying dwell time at a fixed ion group size are given in Figure 3. These confirm the relationship in Figure 2 in showing that decreasing the sampling rate to 1 Hz or less, either by an increase in ion group size or by an increase in dwell time, degrades peak shape. This is especially evident at the longer dwell times for 16-ion groups, where the peak is completely averaged away. Again, oversampling is shown to be ineffective in increasing S/N; as the dwell time is decreased to raise the sampling rate to 3 Hz or greater, only the noise levels are accentuated. This is most apparent in the CCl<sub>4</sub> data for a single ion. Also, it is advantageous to maximize the duty cycle at a fixed sampling rate to improve S/N. This is shown in Figure 3, where data taken for one ion per group with 400-ms dwell are compared with those taken for 16-ion groups at 20-ms dwell. Note that the apparent peak height remains constant with respect to changes in duty cycle; this is a deliberate normalization performed by the ion current acquisition electronics (in the subject mass spectrometers<sup>2</sup>) and cannot be adjusted by the operator. This normalization does not affect the overall S/N improvement with respect to increases in the duty cycle.

#### Conclusions and Recommendations

From these experimental results, it is possible to draw the basic conclusions that SIM mode is inherently more sensitive than scan mode, that various adjustable acquisition parameters affect the resultant S/N, and that similar instrumentation may have widely differing behavior.

Specifically, the following steps should optimize SIM sensitivity:

1. Compartmentalize the total chromatographic run (within software limitations) to minimize the number of ions per group (N).
2. After empirically determining the typical chromatographic peak width (PW) in seconds in a given ion group, calculate the scan rate

(SR) required to achieve 10 data points per peak:

$$SR = 10/PW, \text{ in units of scans per second.}$$

Note that PW is best determined from a chromatographic SIM run performed at a lower limit dwell time (i.e., 20 ms) to ensure that no electronic peak broadening occurs.

3. Calculate the approximate dwell time (DT) in seconds per ion required to achieve the above scan rate for each group:

$$DT = 1/(N \cdot SR), \text{ in units of seconds/ion.}$$

This will be an upper limit because settling times and data acquisition overhead are not yet included.

4. Implement the calculated DTs for each ion group. Perform a trial analysis and determine the actual scan rates (SR[actual]), either from the software display or by closely inspecting the reconstructed chromatogram. Adjust the dwell time to achieve 6 to 10 points per chromatographic peak by choosing a new dwell time for each ion group:

$$DT(\text{new}) < 1/[N \cdot SR(\text{actual})], \text{ in units of seconds/ion.}$$

The existing TO-14 SIM method as used for the TAMS program (Table I) was modified according to this procedure and implemented on the Hewlett-Packard Pascal 3.2 system. The optimization resulted in 10 retention time groups, each with 6 to 8 ions per group. Empirically determined peak widths (including some moderate tailing) ranged from 24 s early in the chromatogram to about 9 s for most of the compounds. The final values for optimized dwell times ranged from 100 to 300 ms/ion to achieve 8 to 10 data points per compound peak.

#### Disclaimer

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