Non-Dietary Exposure Review

Subject:	**EVALUATION OF MIRAN & INTERSCAN FOR MONITORING AIR CONCENTRATIONS OF SULFURYL FLUORIDE**
Guidelines:	
Other:	**Special Review**
DP Barcode:	**D227291**
MRIDs:	
Chemical Codes:	**078003 Sulfuryl fluoride**
Formulation Type:	
Exposed Individual:	
Application Method:	
Outdoor Use Sites:	
Indoor Use Sites:	
Greenhouse Use Sites:	
Other Use Sites:	
Airborne Techniques:	
Dermal Techniques:	
Hand Techniques:	
Foliar Techniques:	
Indoor Surf. Techniques:	
Reviewers:	**David Jaquith**
Review Approvers:	**Sue Hummel Approved on: October 29, 1997**

Sulflur.00

October 29, 19997

MEMORANDUM

SUBJECT: EVALUATION OF MIRAN & INTERSCAN FOR MONITORING AIR CONCENTRATIONS OF SULFURYL FLUORIDE

FROM: David Jaquith
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Please find below the CEB2 review of ....

DP Barcode: D227291 Pesticide Chemical Code: 078003

EPA Reg. No.: 62719-004 MRID No. 438363-01

Deferral to:

PHED: N/A

1.0 INTRODUCTION

CEB2 has been requested to review a study "Evaluation of the MIRAN 101V analyzer and Interscan Model GF1900 VIKANE Analyzer for Determining Sulfuryl Fluoride Concentrations in Air" (MRID No. 438363-00). Sulfuryl Fluoride (SF) is a fumigant used for treatment of structures for dry wood termites, wood boring beetles, cockroaches, and rodents and is marketed under the name VIKANE. The study was submitted by DowElanco in response to a Data Call In notice received by the registrant in November 1993. The study was designed to validate the use of the above two analytical instruments, which are specified on the fumigant label. This study was conducted as part of a larger study measuring levels of SF in the air of treated houses.

2.0 CONCLUSIONS

Examination of the calibration curves for the MIRAN units on the low scale (O - 15 ppm) over the course of the study showed that the MIRAN readings correspond well with the standard concentrations (see Table I for a summary). This is the concentration range of interest for the clearing of structures following fumigations since the clearance level is 5 ppm SF, at which time reentry is allowed without protective equipment. If the calibration data correlated perfectly with the standard concentrations used to generate the calibration curves, the resulting calibration equations would have a slope of 1.0 and pass through the origin. The average slope was 0.972 ($\pm$0.120) and the mean deviation from the origin was 0.141 $\pm$0.093.

The evaluation of the Interscan analyzers focused on the linearity of the instrument response using pre-mixed sulfuryl fluoride standards and the stability of the calibrations over time. The linearity of these instruments was excellent with an average slope of 0.965 (range: 1.01-0.900) and an average deviation from the origin of 0.379 (range: 0.756-0.115 ppm). The coefficient of determination (r^2) of was 1.00. The raw data to verify these calculations were not provided with the report. Deviations between instrument readings and gas bag standards varied with the individual instruments, possibly because of technical difficulties in gas bag preparation. The mean deviations ranged from 14 to 22 percent for the three instruments. At 5 ppm, the concentration at which entrance into the structure is allowed without a self contained breathing apparatus (SCBA), the deviations ranged from 3.2 to 26.4 percent.

The registrant evaluated the stability of the Interscan analyzers over a period of 51 days with long warm-up periods (15 min - 3 hrs) and over a 36 day period when the instruments were allowed to warm-up under more realistic conditions, until the "ready" light came on. These data are summarized in Tables 4-5 and Figures 1-4.

In general the analyzer readings were slightly less than the calibration gas levels. When analyzing calibration gases at concentrations of 4.6 ppm, near the clearance level of 5 ppm when used as a fumigant, the instruments were consistently within approximately 2 ppm of the target concentration for both long and short warm-up scenarios. When the gas standard was 38.9 ppm the instruments were within 10 ppm of the target value. After approximately 28 days the instruments became somewhat erratic, differing from the calibration gases by as much as 80 percent. It must be realized that these particular instruments were maintained in a laboratory environment and not subjected to the rigors that would be expected under normal field use. The registrant did not provide any information on the "typical" calibration pattern used by fumigators. The fact that the registrant was testing the stability of these instruments over time indicates that they are probably not calibrated daily. It would be prudent to calibrate these instruments frequently, at least once per month. A requirement for monthly calibration should be added to the fumigant label.

3.0 DETAILED CONSIDERATIONS

3.1 Materials and Methods

The study was conducted with 2 lots of Sulfuryl Fluoride (VIKANE). The purities of the lots, as analyzed by gas chromatography with a thermal conductivity detector (GC/TCD), were 99.1 and 98.2 percent. The identity of each lot was also verified by gas chromatography/mass spectrometry. Gas standards were also obtained at three concentrations; 38.9 ± 0.4, 4.63 ± 0.05, and 4.64 ± 0.05 ppm, as determined by the supplier. These were used for evaluation of the Interscan analyzers.

3.2 Evaluation of MIRAN 101V:

The MIRAN 101V is a portable infrared analysis unit that detects and measures the fumigant by absorption of infrared light in a predetermined wavelength for SF. Ambient air is continuously pumped through the instrument's air chamber and either percent absorbance or percent transmittance read from a gauge on the unit. The performance of 4 of these units were evaluated in the laboratory prior to their use during the field portion of the house monitoring study. Methods for field calibration were also evaluated.

Laboratory evaluation consisted of the preparation of a series of SF standards in the range of about 0.5 to 8 ppm, followed by analysis with several of the Miran units. A linear calibration plot was developed for each of these units and a linear regression equation developed for each.

The calibration equation was then compared to calibration data obtained from a closed-loop system. The closed-loop system consisted of a stainless steel tee connected to 2 segments of 0.5-inch Tygon tubing. The third arm of the tee was fitted with a septum. The two ends of the Tygon tubing were connected to the inlet and outlet ports of the Miran, forming a continuous loop. The volume of the gas cell in the Miran is known to be 2.5 liters and the volume of the tubing system 0.04 liter. Appropriate volumes of SF were injected into the septum using syringes to obtain nominal concentrations of 0.6 to 8 ppm. The instrument readings were used with the regression equations to determine the SF concentrations, which were then compared with the nominal concentrations.

Because the clearance level for reentry into a treated structure is 5 ppm, the laboratory evaluation of the four MIRAN units prior to their use in the field focused primarily on the linearity of each of the units in the 0.5 - 10 ppm range. Comparison of the gas bag and calibration loop values showed that both methods were in excellent agreement. All of the units were linear over that concentration range with coefficients of determination (r^2) ranging fro m 0.995 to 0.999, with an average and standard deviation of 0.998 ± 0.002. If the two methods agreed perfectly, the slope of a regression of the two methods should yield a slope of 1 and pass through the origin. The comparisons are summarized in Table 1. The raw data required to check calculations were not included in the report.

As previously described, this study was conducted to support an air monitoring study in fumigated homes. The instruments were calibrated extensively at the time of use in the field for each of the houses fumigated. A total of 39 calibration curves were generated in order to convert the analog MIRAN readings taken at each of the time points in the houses to ppm SF. The simplest curve fit that gave a reasonable fit of the calibration line to the data was used in each instance, which often was simply a linear regression. If the calibration data were not linear, a second or third-order polynomial equation had to be used to fit the data reasonably. The use of second and third-order polynomials was only necessary for calibration data generated using the mid (0 - 150 ppm) or high scales (0 - 5000 ppm) of the units (see Figure 2 for a typical mid scale calibration curve). At these concentrations the instrument was probably saturated and outside the linear range. Lack of linearity at these levels does not affect the use of the instrument for clearance purposes. The coefficient of determination (r^2) for all of the calibration equations ranged from 0.960 to 1.00, with an average of 0.995 ± 0.008. This indicates that the calibration equations described the calibration data well.

3.3 Evaluation of Interscan Model GF1900 VIKANE Analyzer

The Interscan Model GF1900 VIKANE Analyzer is the primary field instrument used to approve re-entry into structures fumigated with VIKANE. The analyzer contains

a pyrolyzer that converts sulfuryl fluoride to sulfur dioxide ($SO_2F_2 \rightarrow SO_2$), which is then quantified using an electrochemical sensor. The SF concentration is then shown on an analog display that has a scale of 0 - 50 ppm in 1-ppm increments. Since the clearance value for SF is 5 ppm, much of the study focused on the 0-10 ppm range. The laboratory evaluation of the Interscan units was divided into two portions. The first portion of the evaluation involved three new units obtained from the DowElanco storage warehouse. The instruments were calibrated using a 38.9-ppm SF in air gas standard. The instrument is calibrated by connecting a unit to a gas bag containing the SF in air standard and adjusting the needle to read the appropriate concentration. The unit is re-zeroed and checked against the gas standard several times until a consistent reading is obtained without having to adjust the unit.

An evaluation of the linearity of the units was conducted using both the pre-mixed standard gases and gas bag standards prepared in the laboratory. Gas bag standards were prepared by injecting a known amount of VIKANE into a Tedlar bag containing a known volume of air. Gas bag standards were prepared at nominal concentrations of 1, 3, 5, 7 and 10 ppm SF. At least four readings were taken with the ~5 ppm standards (pre-mixed and gas bag) in order to assess precision. All other readings were typically made in triplicate. The zero of the units were checked against laboratory compressed air between readings and adjusted, if necessary. The linear calibration curves for the pre-mixed standards are presented in Table 2. Due to the inherent variation associated with the preparation of gas bag standards, pre-mixed calibration gases were used as the primary standards to which the gas bag standards were compared. Linearity curves were generated for three of the units using the 4.63 and 38.9 ppm standard gases using the origin as a third concentration. The comparisons between the pre-mixed standard readings and those for gas bag standards are presented in Table 3.

The stability of instrument response was evaluated over the course of 51 days using the pre-mixed gas standards. Portions of the 4.63 and 38.9-ppm commercially-prepared standards were transferred to separate 10-L Tedlar bags and used to determine instrument response at 7 to 8 time points over the course of the 51-day period. All three units were turned on at the same time and allowed to warm up for 45 to 60 minutes prior to making the first reading. By the time that the final reading was taken with the last instrument, that unit had been on for at least two hours. Three to five instrument readings were obtained from the instruments on the day after calibration and at intervals of 3, 8, 17, 28, 35, 45, and 51 days post-calibration. The mean readings, deviation, and percent deviation are presented in Table 4 and the deviation data presented graphically in Figures 1 and 2 for the 4.63 ppm and 38.9 ppm standards, respectively.

The second portion of the instrument stability experiment was conducted to evaluate the stability of instrument response under conditions more typical of use in the field by a fumigator. A fumigator will usually arrive at a house that is to be cleared and immediately power up the Interscan unit. When the "ready" light on the unit illuminates and the unit is put into use. During this portion of the study the readings with the standard gases were made as soon as the "ready" light on the unit was illuminated. The three original units used in the first trial were supplemented by two additional Interscan units (referred to as units 4 and 5). A second ~5 ppm standard gas (4.64 ± 0.05 ppm) was also acquired to evaluate the stability and accuracy of the initial standard gases purchased. All five of the units were calibrated using the 38.9-ppm standard gas prior to initiation of the experiment. Instrument response was evaluated at 7 time points over a 36 day period. These data are summarized in Table 5 and the deviations graphically in Figures 3 and 4 for the 4.63-4.64 and 38.9 ppm standards, respectively.

Table 1. Summary of Low Scale (O - 15 ppm Sulfuryl Fluoride) MIRAN 101V Analyzer Field Calibration Curve Data.

MIRAN Serial Number	Date of Fumigation	Deviation from Origin (ppm)	Slope	Average Deviation from Target (%)
001	3/15/94	0.280	0.931	14.2
306	3/16/94	0.007	1.050	6.05
001	3/17/94	0.248	0.959	12.4
306	3/18/94	0.064	0.989	4.88
266	3/19/94	0.105	0.861	4.29
306	3/20/94	0.042	0.953	7.81
266	3/21/94	0.134	0.791	14.9
266	3/99/94	0.026	0.720	9.21
306	4/11/94	0.188	1.050	9.99
001	4/12/94	0.269	0.806	14.1
306	4/13/94	0.118	1.030	4.65
306	4/14/94	0.024	1.060	7.43
101-304V	4/14/94	0.177	1.090	5.87
101-304V	4/15/94	0.254	1.110	8.29
306	4/18/94	0.204	1.080	8.16
101-304V	4/19/94	0.109	1.070	7.17
Mean		**0.141**	**0.972**	**8.71**
Standard Deviation		**0.093**	**0.120**	**3.50**

from report ECL-94013a (1). Complete agreement should have a slope of 1.0 and pass through the origin.

Table 2. **Summary of Linear Calibration Curves for Interscan GF1900 VIKANE Analyzers Using Pre-Mixed Calibration Gases.**

Interscan Number	Slope	Deviation from Origin (ppm)	r^2
1	0.984	0.756	0.999
2	0.900	0.115	1.00
3	1.01	0.265	1.00
Mean	**0.965**	**0.379**	**1.00**
Std. Deviation	**0.057**	**0.335**	**0.00058**

Table 3. Summary of the Comparison of Gas Bag Standards to Interscan GF1900 VIKANE Analyzer Linearity Curve Generated Using Pre-Mixed Gas Standards.

Nominal Bag Concentration[1] (ppm)	Absolute Value of Deviation from Nominal SF Concentration (%)		
	Unit I	Unit 2	Unit 3
1	80.0	20.0	10.0
1	60.0	0.0	10.0
1	80.0	0.0	10.0
Mean for Instrument	**73.3**	**6.7**	**10.0**
3	13.3	23.3	26.7
3	6.7	23.3	26.7
3	6.7	13.3	26.7
Mean for Instrument	**8.9**	**20.0**	**26.7**
5	4.0	14.0	32.0
5	4.0	14.0	28.0
5	0.0	14.0	24.0
5	4.0	14.0	24.0
5	4.0	14.0	24.0
Mean for Instrument	**3.2**	**14.0**	**26.4**
7	10.0	15.7	22.9
7	10.0	18.6	22.9
7	10.0	18.6	22.9
Mean for Instrument	**10.0**	**17.6**	**22.9**
10	3.0	10.0	20.0
10	3.0	10.0	18.0
10	5.0	10.0	20.0
Mean for Instrument	**3.7**	**10.0**	**19.3**
Mean for All Instruments	**18**	**14**	**22**
Standard Deviation for All Instruments	**27**	**6.6**	**6.5**

[1] **The term nominal refers to the concentration of sulfuryl fluoride prepared in the Tedlar bag as discussed in the text.**

Table 4. Summary of the Mean Instrument Readings of Three Interscan Instruments At Intervals Over a 51-Day Period After Calibration with Sulfuryl Standards.

Instr. No.	Day After Calibration	Mean Reading (ppm)[1]		Mean Dev. (ppm)		Mean Dev. (%)	
		4.63 ppm Std.	38.9 ppm Std.	4.63 ppm Std.	38.9 ppm Std.	4.63 ppm Std.	38.9 ppm Std.
1	1	ND[2]	ND	ND	ND	ND	
	3	3.6	37.7	-1	-1.2	-21.6	-3.1
	8	3.1	27.7	-1.5	-11.2	-32.4	-28.8
	17	2.9	25.8	-1.7	-13.1	-36.7	-33.7
	28	2.8	26.3	-1.8	-12.6	-38.9	-32.4
	35	3.6	32.7	-1	-6.2	-21.6	-15.9
	45	2.5	19.7	-2.1	-19.2	-45.4	-49.4
	51	3.4	34	-1.2	-4.9	-25.9	-12.6
	51R[2]	2.6	24.3	-2	-14.6	-43.2	-37.5
	51R2[2]	2.4		-2.2		-47.5	
2	1	ND	ND	ND	ND	ND	ND
	3	4	35	-0.6	-3.9	-13	-10
	8	3.3	33	-1.3	-5.9	-28.1	-15.2
	17	2.8	29	-1.8	-9.9	-38.9	-25.4
	28	2.7	28.3	-1.9	-10.6	-41	-27.2
	35	2.8	28.7	-1.8	-10.2	-38.9	-26.2
	45	2	20.7	-2.6	-18.2	-56.2	-46.8
	51	1	9	-3.6	-29.9	-77.8	-76.9
	51R	0.8	6.8	-3.8	-32.1	-82.1	-82.5

Table 4. Summary of the Mean Instrument Readings of Three Interscan Instruments At Intervals Over a 51-Day Period After Calibration with Sulfuryl Standards.

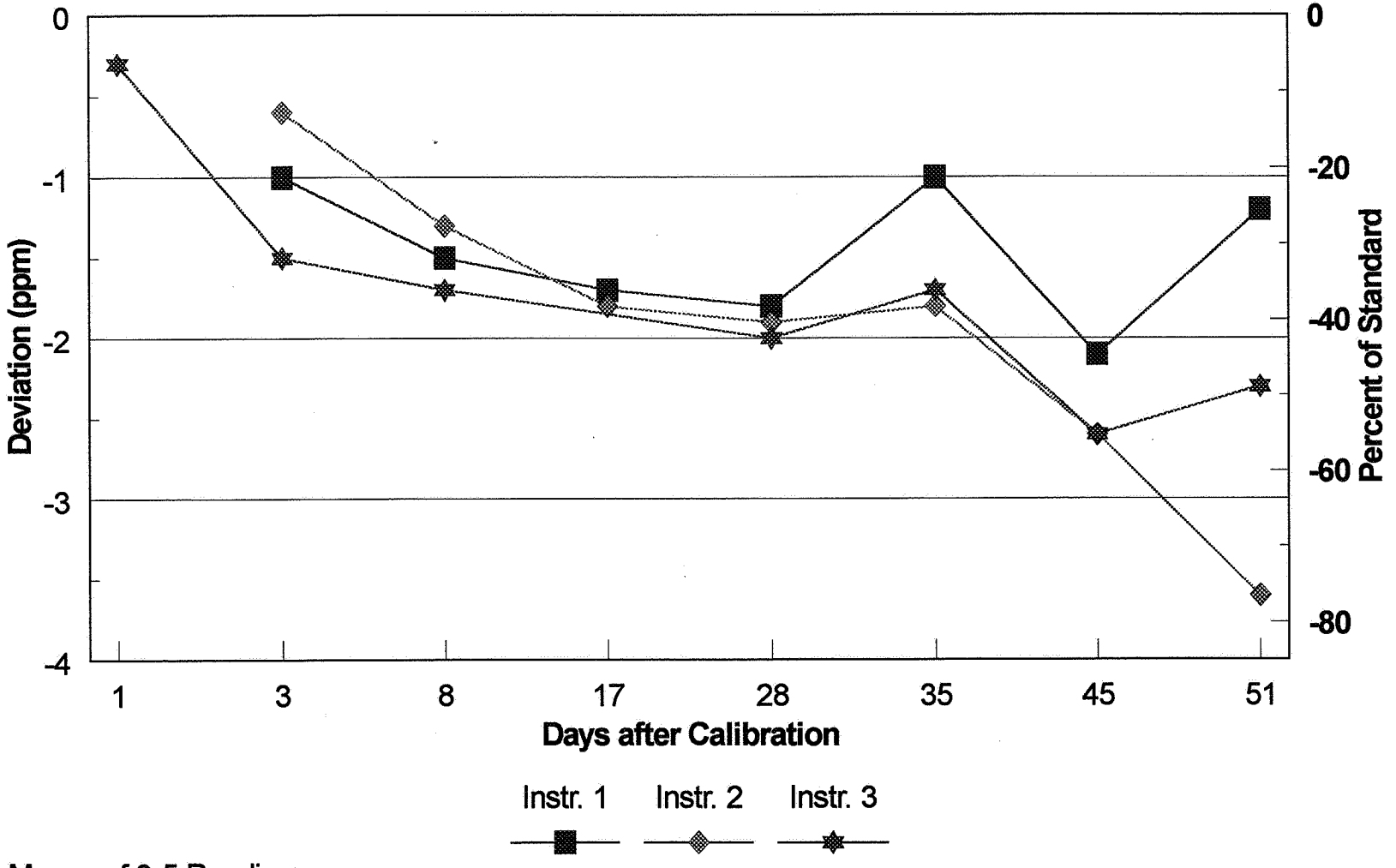
Instr. No.	Day After Calibration	Mean Reading (ppm)[1]		Mean Dev. (ppm)		Mean Dev. (%)	
		4.63 ppm Std.	38.9 ppm Std.	4.63 ppm Std.	38.9 ppm Std.	4.63 ppm Std.	38.9 ppm Std.
3	1	4.3	39.1	-0.3	0.2	-6.5	0.5
	3	3.1	ND	-1.5	ND	-32.4	
	8	2.9	34.8	-1.7	-4.1	-36.7	-10.5
	17	ND	28.7	ND	-10.2	ND	-26.2
	28	2.6	26.7	-2	-12.2	-43.2	-31.4
	35	2.9	28	-1.7	-10.9	-36.7	-28
	45	2	21	-2.6	-17.9	-56.2	-46
	51	2.3	23	-2.3	-15.9	-49.7	-40.9

[1] Values are the mean of 3-5 separate readings.

[2] ND = No Data

[2] Days labeled 51R and 51R2 are repeat confirmatory readings on that day because of erratic or low measurements.

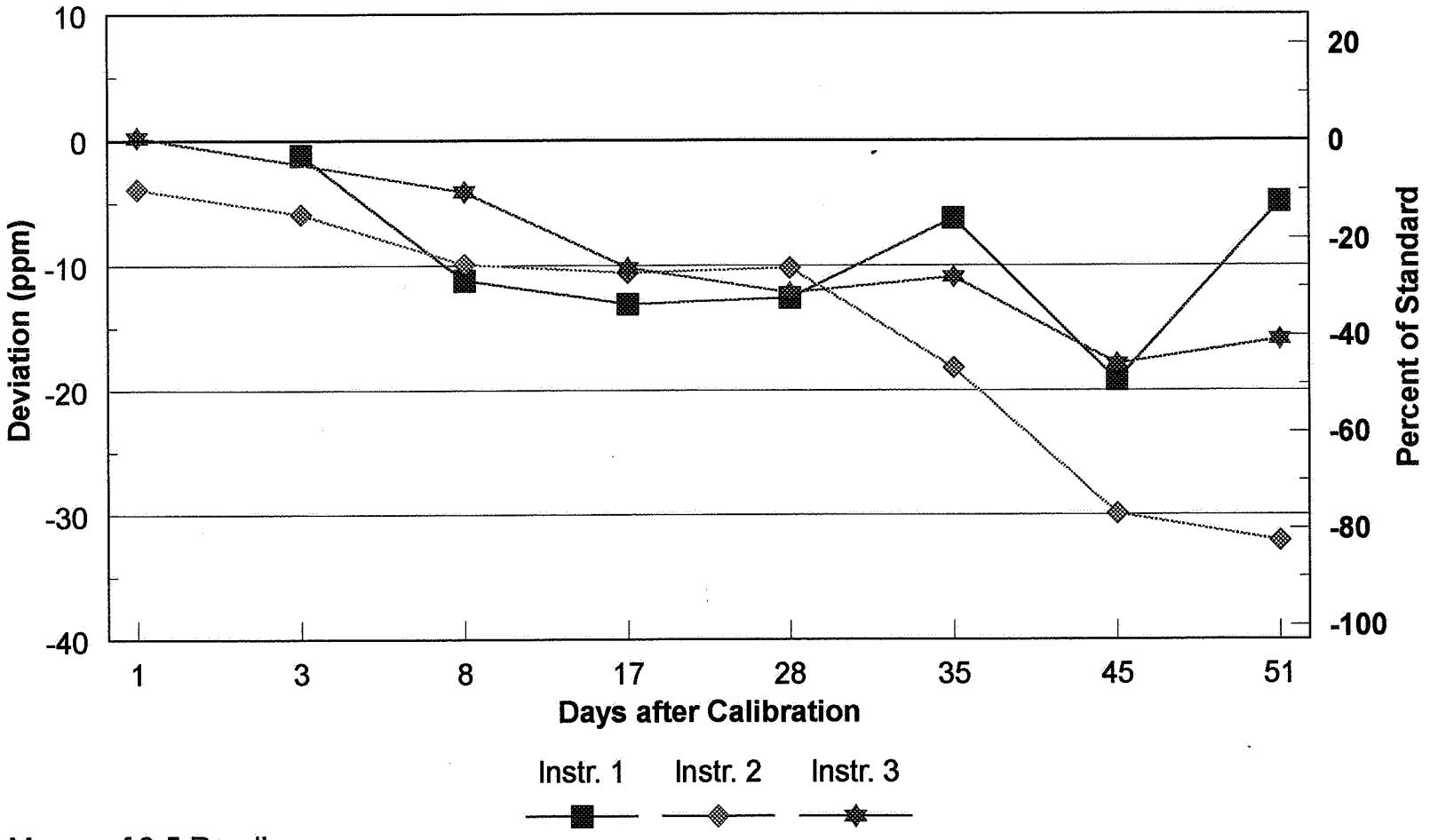
Figure 1. Deviations of Interscan Analyzer Readings from a 4.63 ppm Sulfuryl Fluoride Standard Over a 51-Day Period.



Means of 3-5 Readings

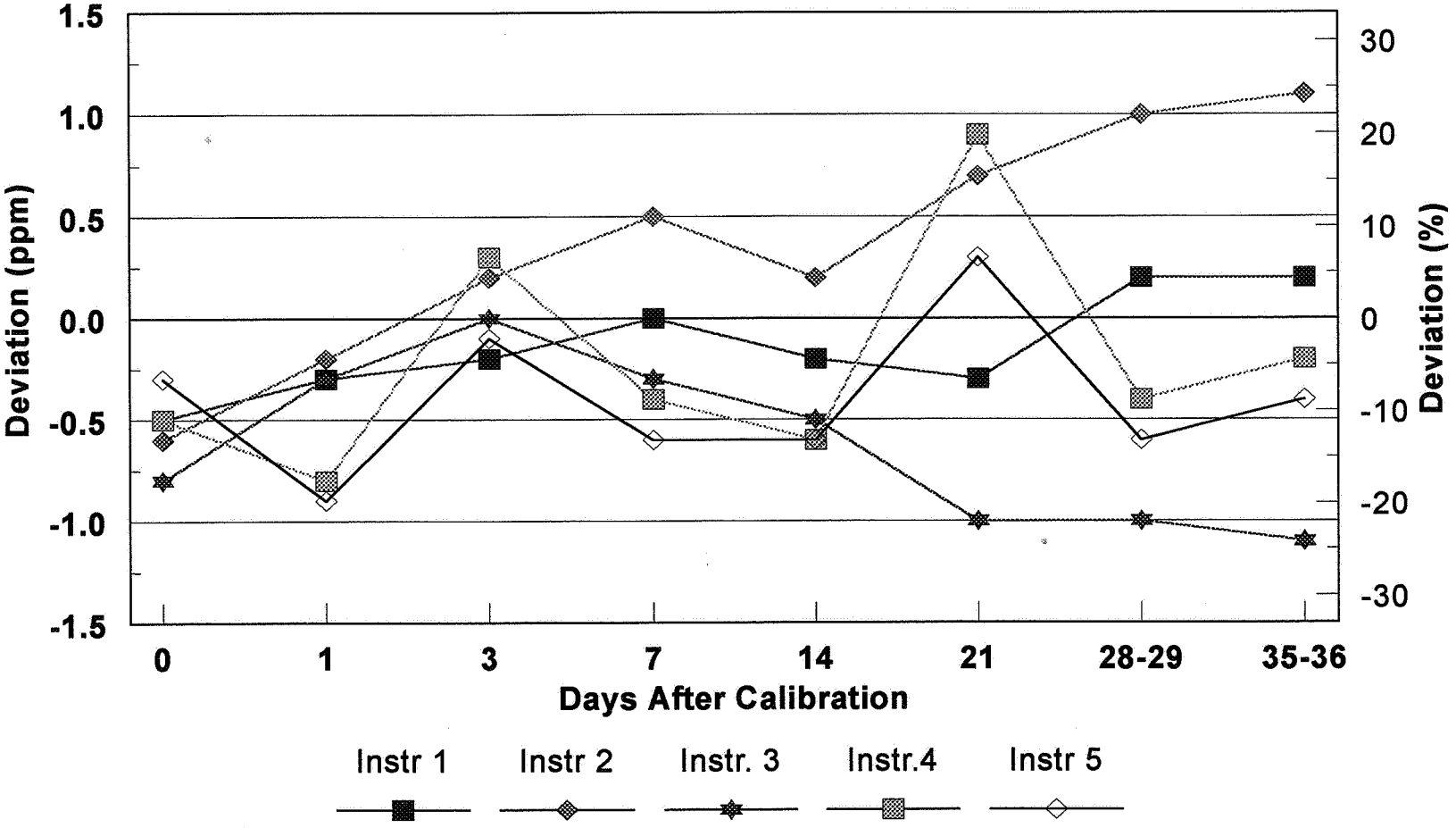
Instruments were allowed to equilibrate for 15-min to 3 hours prior to use.

Figure 2. Deviations of Interscan Analyzer Readings from a 38.9 ppm Sulfuryl Fluoride Standard Over a 51-Day Period.



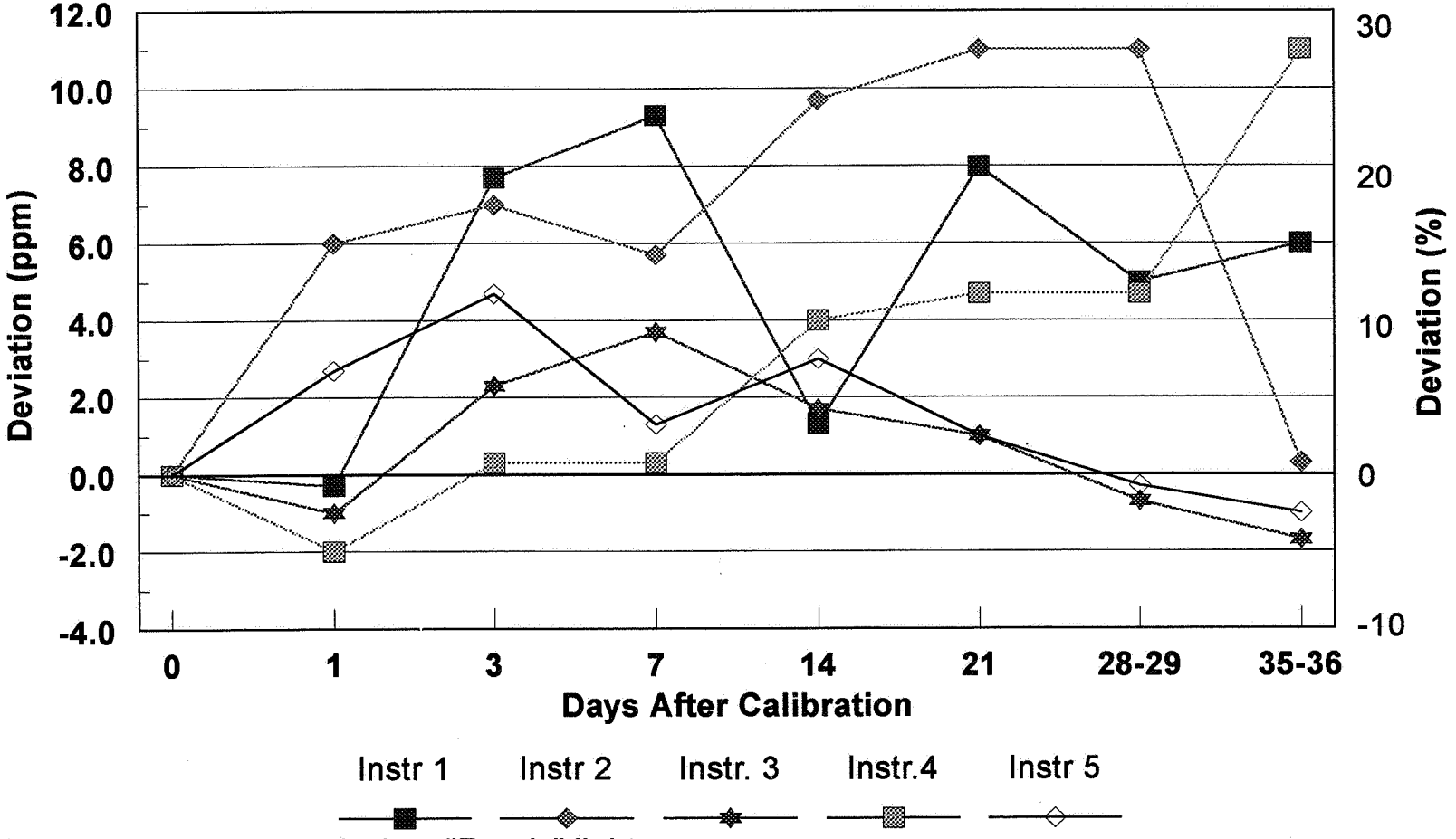
Means of 3-5 Readings.
Instruments were allowed to equilibrate for 15-min to 3 hours prior to use.

Figure 3. Deviations of Interscan Analyzer Readings from 4.63-4.64 ppm Sulfuryl Fluoride Standards Over a 36-Day Period Following Mini Minimal Warm-UP.



Mean of 6 Readings.
Instruments were used when "Ready" light came on.

Figure 4. Deviations of Interscan Analyzer Readings from a 38.9 ppm Sulfuryl Fluoride Standard Over a 36-Day Period Following Minimal Warm-Up



Instruments were used when "Ready" light came on.
Mean of 3 Readings.

Table 5. Summary of Readings and Deviations of Interscan Analyzers from Pre-Mixed Sulfuryl Fluoride Standards Over a 36-Day Period Following Minimal Warm-up. [1]

Instrument No.	Days Since Calibrated	Standard Concentration (ppm)	Meter Reading (ppm)	Deviation (ppm)	Deviation (%)
1	0	4.6	4.1	-0.5	-11.5
2	0	4.6	4.0	-0.6	-13.3
3	0	4.6	3.8	-0.8	-17.3
4	0	4.6	4.1	-0.5	-11.5
5	0	4.6	4.3	-0.3	-5.7
1	1	4.6	4.3	-0.3	-6.5
2	1	4.6	4.4	-0.2	-4.3
3	1	4.6	4.3	-0.3	-7.2
4	1	4.6	3.8	-0.8	-17.3
5	1	4.6	3.7	-0.9	-19.4
1	3	4.6	4.5	-0.2	-3.2
2	3	4.6	4.8	0.2	4.7
3	3	4.6	4.6	0.0	0.0
4	3	4.6	4.9	0.3	6.5
5	3	4.6	4.5	-0.1	-2.9
1	7	4.6	4.6	0.0	0.7
2	7	4.6	5.1	0.5	10.0
3	7	4.6	4.3	-0.3	-6.5
4	7	4.6	4.2	-0.4	-9.3
5	7	4.6	4.0	-0.6	-12.2
1	14	4.6	4.4	-0.2	-4.7
2	14	4.6	4.8	0.2	5.1
3	14	4.6	4.1	-0.5	-10.4
4	14	4.6	4.0	-0.6	-13.7
5	14	4.6	4.0	-0.6	-12.2
1	21	4.6	4.3	-0.3	-5.7
2	21	4.6	5.3	0.7	15.1
3	21	4.6	3.6	-1.0	-20.9
4	21	4.6	5.5	0.9	20.2
5	21	4.6	4.9	0.3	5.7

[1] Values are means of 3-6 readings. The 4.6 ppm standard includes both 4.63 and 4.64 ppm standards.

Table 5. Summary of Readings and Deviations of Interscan Analyzers from Pre-Mixed Sulfuryl Fluoride Standards Over a 36-Day Period Following Minimal Warm-up. [1]

Instrument No.	Days Since Calibrated	Standard Concentration (ppm)	Meter Reading (ppm)	Deviation (ppm)	Deviation (%)
1	28	4.6	4.8	0.2	3.6
2	28	4.6	5.6	1.0	21.9
3	28	4.6	3.6	-1.0	-22.3
4	29	4.6	4.2	-0.4	-7.9
5	29	4.6	4.0	-0.6	-12.2
4	35	4.6	4.4	-0.2	-3.6
5	35	4.6	4.2	-0.4	-9.4
1	36	4.6	4.8	0.2	5.1
2	36	4.6	5.7	1.1	24.5
3	36	4.6	3.6	-1.1	-22.7
1	0	38.9	39	0	0.0
2	0	38.9	39	0	0.0
3	0	38.9	39	0	0.0
4	0	38.9	39	0	0.0
5	0	38.9	39	0	0.0
1	1	38.9	39	-0	-0.9
2	1	38.9	45	6	15.4
3	1	38.9	45	6	15.4
4	1	38.9	38	-1	-2.6
5	1	38.9	37	-2	-5.1
1	3	38.9	42	3	6.8
2	3	38.9	47	8	19.7
3	3	38.9	46	7	18.0
4	3	38.9	41	2	6.0
5	3	38.9	39	0	0.9
1	7	38.9	44	5	12.0
2	7	38.9	48	9	24.0
3	7	38.9	45	6	14.6
4	7	38.9	43	4	9.4
5	7	38.9	39	0	0.9

[1] Values are means of 3-6 readings. The 4.6 ppm standard includes both 4.63 and 4.64 ppm standards.

Table 5. Summary of Readings and Deviations of Interscan Analyzers from Pre-Mixed Sulfuryl Fluoride Standards Over a 36-Day Period Following Minimal Warm-up. [1]

Instrument No.	Days Since Calibrated	Standard Concentration (ppm)	Meter Reading (ppm)	Deviation (ppm)	Deviation (%)
1	14	38.9	40	1	3.4
2	14	38.9	49	10	24.8
3	14	38.9	41	2	4.3
4	14	38.9	43	4	10.3
5	14	38.9	40	1	2.6
1	21	38.9	42	3	7.7
2	21	38.9	50	11	28.3
3	21	38.9	40	1	2.6
4	21	38.9	44	5	12.0
5	21	38.9	40	1	2.6
1	28	38.9	44	5	12.9
2	28	38.9	50	11	28.3
3	28	38.9	38	-1	-1.7
4	29	38.9	44	5	12.0
5	29	38.9	39	-0	-0.9
4	35	38.9	45	6	15.4
5	35	38.9	39	0	0.9
1	36	38.9	37	-2	-4.3
2	36	38.9	50	11	28.3
3	36	38.9	38	-1	-2.6

[1] Values are means of 3-6 readings. The 4.6 ppm standard includes both 4.63 and 4.64 ppm standards.

REFERENCES

1) Shurdut, B. A., Murphy, P. G. and K. K. Beard, Evaluation of Concentration of Sulfuryl Fluoride Inside Houses Following Fumigation with VIKANE Gas Fumigant, Global Human Exposure Assessment, DowElanco, Indianapolis, IN, Report ECL-94013,1994.

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Sulfuryl fluoride file/078003
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