(12)/cw/14/23

2001 NASA/ASEE SUMMER FACULTY FELLOWSHIP PROGRAM

JOHN F. KENNEDY SPACE CENTER
UNIVERSITY OF CENTRAL FLORIDA

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN A CONTROLLED ENVIRONMENT: ETHYLENE GAS MEASUREMENT STUDIES ON RADISH

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ABSTRACT

Volatile organic compound(VOC), ethylene gas, was characterized and quantified by GC/FID. 20-50 ppb levels were detected during the growth stages of radish. SPME could be a good analytical tool for the purpose. Low temperature trapping method using dry ice/diethyl ether and liquid nitrogen bath was recommended for the sampling process for GC/PID and GC/MS analysis.

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1. INTRODUCTION

Volatile organic compound(VOC), ethylene gas, was characterized and quantified by Gas Chromatograpny-Flame Ionization Detector (GC/FID). Radish was grown in a controlled environment. . Raising plants in a closed environment could be one of the challenges in space. In such a system, the possibilities present for some VOCs to reach levels that could lead to poor plant growth. Ethylene level of 20-50 ppb was detected during the growth stages of radish. For all tests, the materials used for the experiment (e,g, vinyl tubing, plastic cups, and epoxy resins) were not significant sources for ethylene but needed to be addressed. Methods using GC/MS, GC/PID, GC/FID and SPME(solid phase microextraction) to measure the VOCs have been studied from planting to harvest, under controlled environmental atmosphere.

2. Experimental Methods

1). Planting the seeds.

Cherry Bell Radish was purchased from W. Atlee Burpee & Co., Warminster, PA. The radish has a round shape, smoth, redi color, white flesh and ready 22 days after seeding to harvest. It was recommended to sow in average stone-free soil in early to late spring and again in late summer. In Deep South, Gulf and Pacific Coast areas, sow from fall to early spring. In rows 6" or more apart, sow seeds evenly and cover with ½" of fine soil. Firm lightly. Keep soil most. Seedlings emerge in 7-10 days depending on soil and weather conditions. It thrives in cool weather. Recommended to jake succe3ssive plantings every 2 weeks until late spring then again a moth before frost.

six seeds were placed in a Oasis foam (medium growing foam) saturated with Hoggland's solution in a 3x3 " magenta (outside covered with black electrical masking tape) container. There are 6 duplications and a control.

2). **Ethylene studies**

The schedule for the experiement is as follow:

June 29th.	Plant seed.	
July 6th.	1st thinning.	7 days after.
9th.	Dry weight check after 72 hours.	
July 13th.	2nd thinning.	14 days after.
16th.	Dry weight check after 72 hours.	
July 20th.	Harvest.	21 days after.
23rd.	Dry weight check after 72 hours.	

3. **Method for Ethylene**

In a Percival chamber, 3 radish jars were placed. The chamber is supplied with lights, 3000 ppm of Carbon dioxide gas. Humidity is controlled. The plants were transferred in a bell jar every day for ethylene studies. The air samples were collected 1, 2, and 3 hours interval. The plants were moved out of the bell jar after sampling. There was a control pot with the same conditions without seed.
Ethylene gas sample was collected with syringe.

Gas chromatography-Flame Ionization Detector(GC/FID) was used to measure the emission rate. Gas chromatography-Mass Spectrometer (GC/MS), Gas chromatography-Photoionization Detector (GC/PID) and Solid Phase Microextraction (SPME) sampling techniques were also investigated.

Figure 1. Bell jar and cooling system inside of the growth chamber.

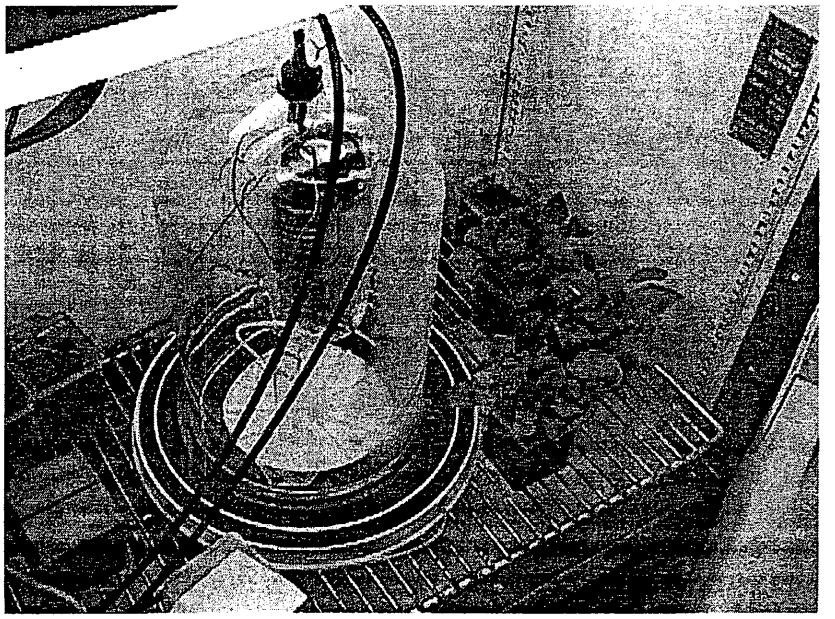


Figure 2. Plant growth after 20 days.

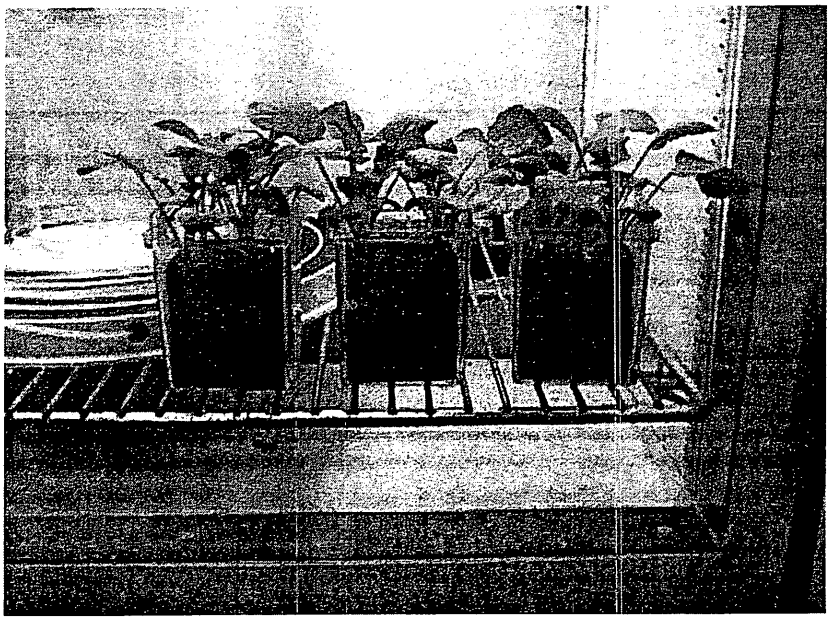
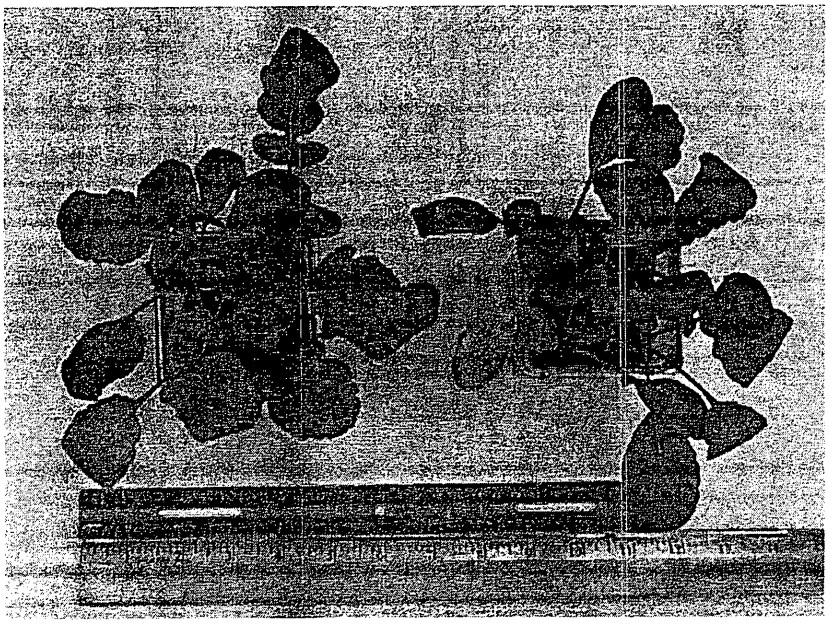


Figure 3. Leaf area measurement.



4. Results

Ethylene gas emission rate was measured with Gas Chromatography-Flame Ionization Detector (GC-FID).

Detectable Limits = 3(Noise): signal
Noise = 0.058825
Detectable Limits (PPB) = 11.80

Analysis Date	Sample Date	Sample File Name	Ethylene	% SD	Peak Area
7/11/01	7/6/01	control	28.392		0.436
7/11/01	7/6/01	control	11.596		0.191
7/11/01	7/6/01	1 hour	32.004		0.489
7/11/01	7/6/01	1 hour	21.400		0.334
7/11/01	7/6/01	2 hours	14.201		0.229
7/11/01	7/6/01	2 hours	12.842		0.209
7/11/01	7/6/01	3 hours	13.003	0.83%	0.212
7/11/01	7/6/01	3 hours	13.173		0.214
7/11/01	7/9/01	control	17.492		0.277
7/11/01	7/9/01	control	14.750		0.237
7/11/01	7/9/01	1 hour	22.908	0.60%	0.356
7/11/01	7/9/01	1 hour	22.702		0.353
7/11/01	7/9/01	2 hours	21.537	1.47%	0.336
7/11/01	7/9/01	2 hours	21.057		0.329
7/11/01	7/9/01	3 hours	29.900	0.15%	0.458
7/11/01	7/9/01	3 hours	29.832		0.457

Result. Continued.

Detectable Limits = 3(Noise): signal
Noise = 0.058825
Detectable Limits (PPB) = 11.80

Analysis Date	Sample Date	Sample File Name	PPB Ethylene	% SD	Peak Area
7/16/01	7/10/01	control	24.622	1.99%	0.381
7/16/01	7/10/01	control	23.888		0.370
7/16/01	7/10/01	1 hour	27.021	0.00%	0.416
7/16/01	7/10/01	1 hour	27.021		0.416
7/16/01	7/10/01	2 hours	27.844	0.50%	0.428
7/16/01	7/10/01	2 hours	27.638		0.425
7/16/01	7/10/01	3 hours	34.631	1.88%	0.527
7/16/01	7/10/01	3 hours	33.671		0.513
7/16/01	7/11/01	control	39.292	0.00%	0.595
7/16/01	7/11/01	control	39.292		0.595
7/16/01	7/11/01	1 hour	46.354	0.61%	0.698
7/16/01	7/11/01	1 hour	45.942		0.692
7/16/01	7/11/01	2 hours	42.096	0.23%	0.636
7/16/01	7/11/01	2 hours	42.240		0.638
7/16/01	7/11/01	3 hours	39.704	#DIV/0!	0.601
7/16/01	7/11/01	3 hours	<2.51		
7/17/01	7/13/01	control	36.345	0.77%	0.552
7/17/01	7/13/01	control	36.758		0.558
7/17/01	7/13/01	1 hour	33.280		0.507
7/17/01	7/13/01	1 hour	32.300		0.493
7/17/01	7/13/01	2 hours	38.744	0.48%	0.587
7/17/01	7/13/01	2 hours	38.470		0.583
7/17/01	7/13/01	3 hours	42.448	0.11%	0.641
7/17/01	7/13/01	3 hours	42.377		0.640

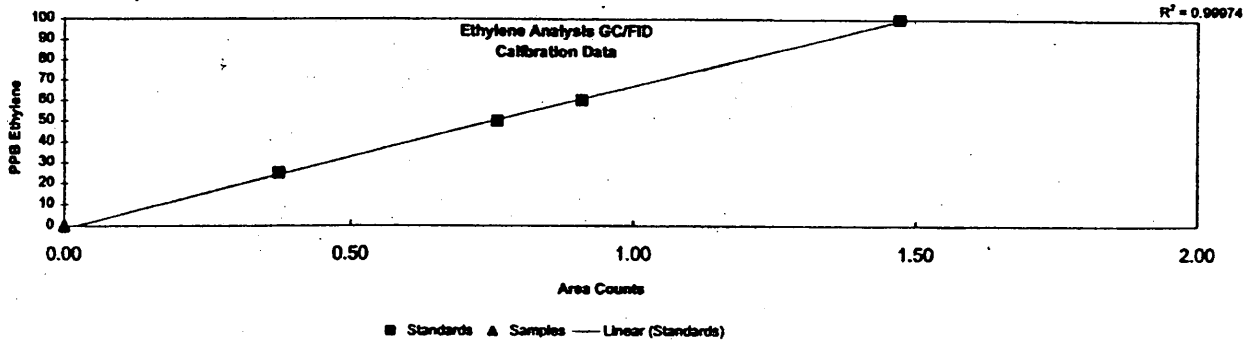
Figure 3. Calibration curve.

Ethylene Analysis

06/19/01
BPS MVT
Barbara Peterson: Organic Analytical Lab
Data Reference: Chem/Orbitac/BPS MVT

Ethylene Analysis

			Standards	PPB Ethylene	Peak Area	Response Factor	
			4/17/01	25	0.374	66.8548	
				50	0.760	65.7485	
				60	0.910	65.9424	
				100	1.471	67.9810	
HP 6890	GC/FID			Continuing Calibration			
FID	350	Degrees Centigrade		100	1.456	99.0%	07/03/01
FID-O2	100	mL/Minute Oxygen		100	1.474	100.2%	07/05/01
FID-H2	32	mL/Minute Hydrogen		100	1.460	99.2%	07/06/01
Oven	20(@10->54(1.6)	Degrees Centigrade		100	1.460	100.6%	07/09/01
Carrier	6.6	mL/Minute Helium		100	1.457	99.0%	07/11/01
				100	1.460	99.3%	07/14/01
				100	1.475	100.3%	07/16/01
				100	1.468	99.7%	07/17/01
Rt	4.159	minutes		Regression		25-100 PPB	
Run Time	5	minutes		Constant		-1.497	
Column	0.54mm X 30M HP Plot Q			Std Err of Y Est		1.084	
				R Squared		0.99960	
				No. of Observations		4	
				X Coefficient(s)		68.555	



5. **Conclusions**

1. Well Calibrated Photovac (GC/Photo Ionization Detector) or GC equipped with Flame Ionization Detector (FID) could be more useful to detect ethylene gas in the chamber if a longer GC column is used or temperature programmed.
2. Solid Phase Microextraction (SPME) applied to GC/MS will be an interesting sampling device to try if quantification and calibration are not that time consuming. The present gas sampling method needs to be improved.
3. Sample concentration process is recommended.
4. Trapping the gas in low temperature with diethyl dry ice bath and liquid nitrogen could separate the ethylene gas from carbon dioxide and nitrogen. It can produce better spectra minimizing the interferences resulting a good GC/PID and GC/MS resolution.
5. Further studies on this subject are necessary.

6. **References**

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Acknowledgements

The author wished to thank Drs, Gary Stutte, Barbara Peterson, Lanfang Levine, Greg Goins, Jay Garland, Vadim Rygalov, Kathleen Daumer and Ignacio Eraso, Dynamac Corporation, and Dr. Raymond Wheeler, National Aeronautic and Space Administration Biological and Life Support, Kennedy Space Center, FL 32899, U.S.A for the time, energy and efforts that helped my tour of duty. The author also would like to thank the summer university faculty program officers and staff members and researchers at Kennedy Space Center, NASA and University of Central Florida.