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# **Reproducibility of Responses in Polymer-Carbon Composite Films in an Electronic Nose Sensing Array**

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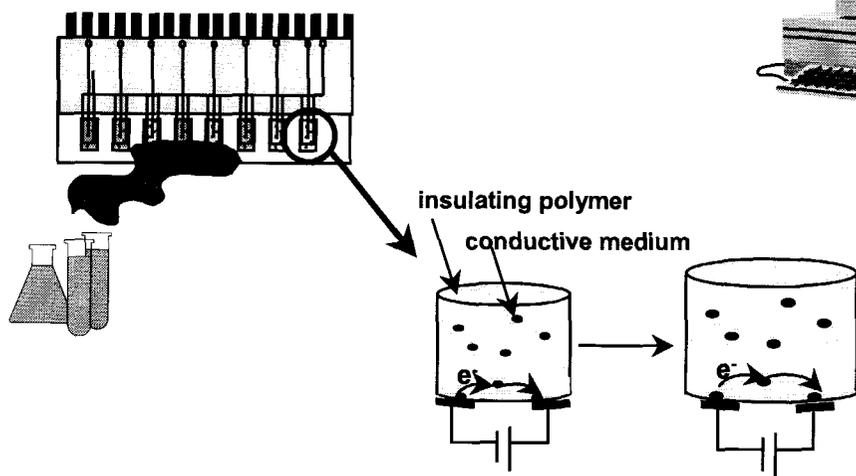
**JPL**



An array of non-specific chemical sensors, controlled and analyzed electronically, which mimics the action of the mammalian nose by recognizing patterns of response

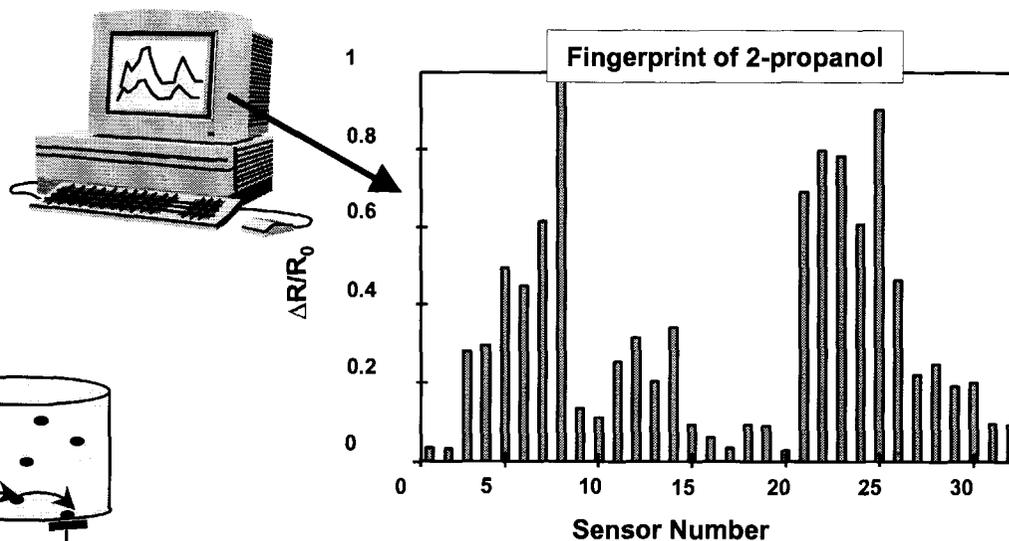
1. ENose measures background resistance in each sensor and establishes  $R_0$ .

2. Contaminant comes in contact with sensors on the sensing head.



3. The sensing media, polymer films loaded with a conductive medium such as carbon black, change resistance by swelling or shrinking as air composition changes.

4. Resistance is recorded by a computer, the change in resistance is computed, and the distributed response pattern of the sensor array is used to identify gases and mixtures of gases



5. Responses of the sensor array are analyzed and quantified using software developed for the task.

**propanol 300 ppm**

## ***LONG-DURATION SPACE FLIGHT***

- ☞ **High level of crew productivity; little habitat maintenance**
- ☞ **Decouple environmental control from ground control**
  - ⊙ **Distributed network of sensors and actuators**
  - ⊙ **Sound an alarm and/or actuate remedial action**
  - ⊙ **Early identification of areas requiring remediation**

## ***ELECTRONIC NOSE***

- ☞ **Incident monitor for contaminants exceeding Spacecraft Max Allowable Concentration (SMAC)**
- ☞ **Monitor clean-up process**



# Electronic Nose ELECTRONIC NOSE FOR SPACE STATION



## **ELECTONIC NOSE:**

- ◆ Incident monitor - real time air quality monitoring
- ◆ Identify and quantify target compounds at SMAC level
- ◆ Low mass, low power device
- ◆ Requires little crew time for maintenance and calibration

	<b>Analysis Time (min)</b>	<b>Concentration of Constituents</b>	<b>Discrimination of Constituents</b>
<b>ENose</b>	<b>.5 - 15</b>	<b>0.01 - 10,000 ppm</b>	<b>good for target set</b>
<b>GC-MS</b>	<b>10 - 100</b>	<b>&lt; 10 ppb</b>	<b>very good</b>
<b>Optical</b>	<b>1 - 5</b>	<b>0.01 - 2000 ppm</b>	<b>good for target set</b>
<b>VOC</b>	<b>1 - 5</b>	<b>.1 - 2000 ppm</b>	<b>poor</b>
<b>FID</b>	<b>1 - 5</b>	<b>.1 - 50,000 ppm</b>	<b>poor</b>
<b>Smoke Alarm</b>	<b>.5 - 5</b>	<b>1 - 10 ppb</b>	<b>none</b>



*Electronic Nose*  
**TARGET COMPOUNDS**



<i>Phase I</i>		<i>Phase II</i>	
<b>Compound</b>	<b>24 hr SMAC (ppm)</b>	<b>Compound</b>	<b>24 hr SMAC (ppm)</b>
ammonia	20	acetaldehyde	6
benzene	3	acetone	270
ethanol	500	acetonitrile	4
formaldehyde	0.1	2-butanone	150
Freon 113	50	chlorobenzene	10
indole	0.3	dichloromethane	35
methane	5300	furan	0.1
methanol	10	hexamethylcyclo-	
2-propanol	100	trisiloxane	25
toluene	16	hydrazine	0.3
		methyl hydrazine	0.002
		tetrahydrofuran	40
		1,1,1-trichloroethane	11
		xylene	100



## ***REPRODUCIBILITY and REPEATABILITY***

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### **◆ SENSORS - *REPRODUCIBILITY***

**Two sensors made from same solution (same/different person)**

- ❖ **Baseline resistance**
- ❖ **Noise level**
- ❖ **Response to an analyte**

**Two sensors made from different solutions (same/different person)**

- ❖ **Baseline resistance**
- ❖ **Noise level**
- ❖ **Response to an analyte**

### **◆ SENSORS - *REPEATABILITY***

- ❖ **Response to an analyte delivered several times in any order**
- ❖ **Noise level**

***REPRODUCIBILITY and REPEATABILITY***

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**◆ ARRAY - *REPRODUCIBILITY***

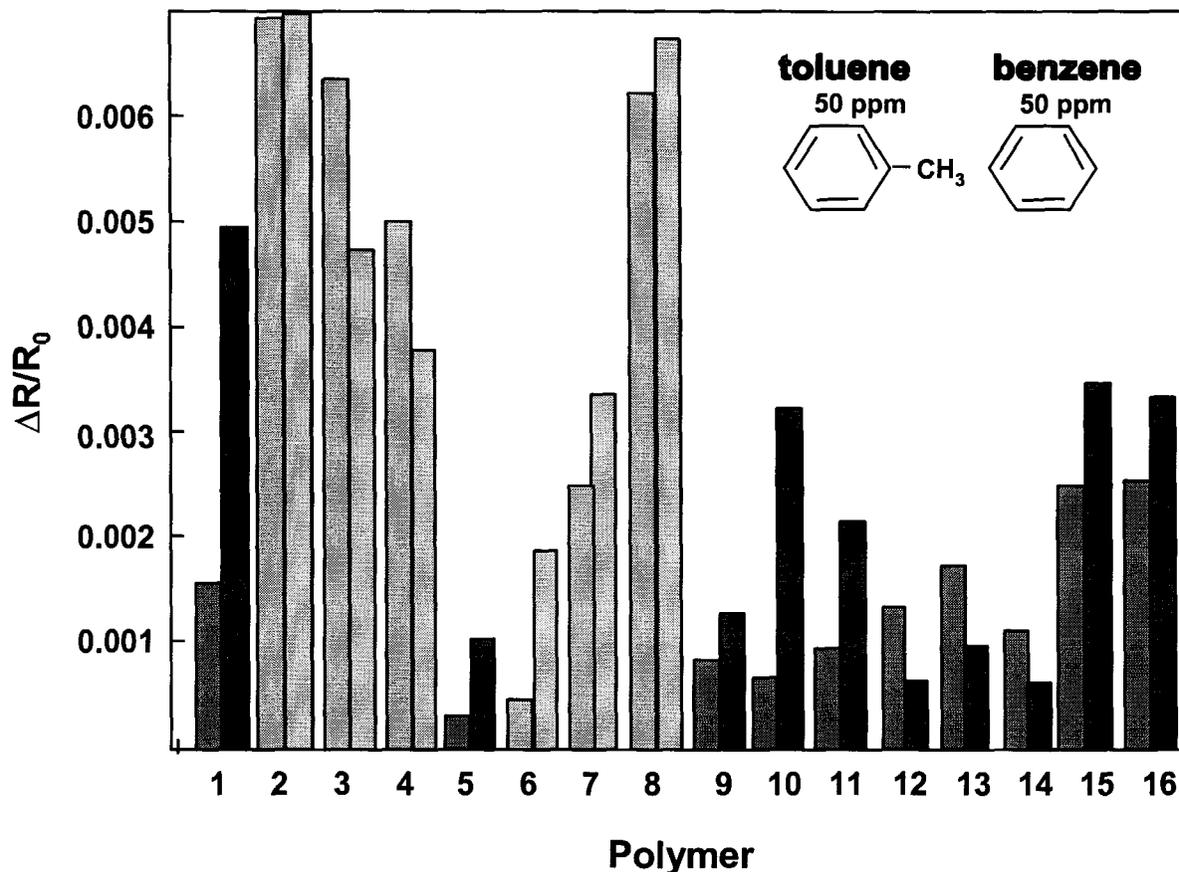
- ❖ Depends on Sensor Reproducibility
- ❖ Required for use of single software training set without extensive recalibration

**◆ ARRAY - *REPEATABILITY***

- ❖ Identification software can repeatedly identify and quantify an analyte
- ❖ Repeated stimulus can be delivered in any order

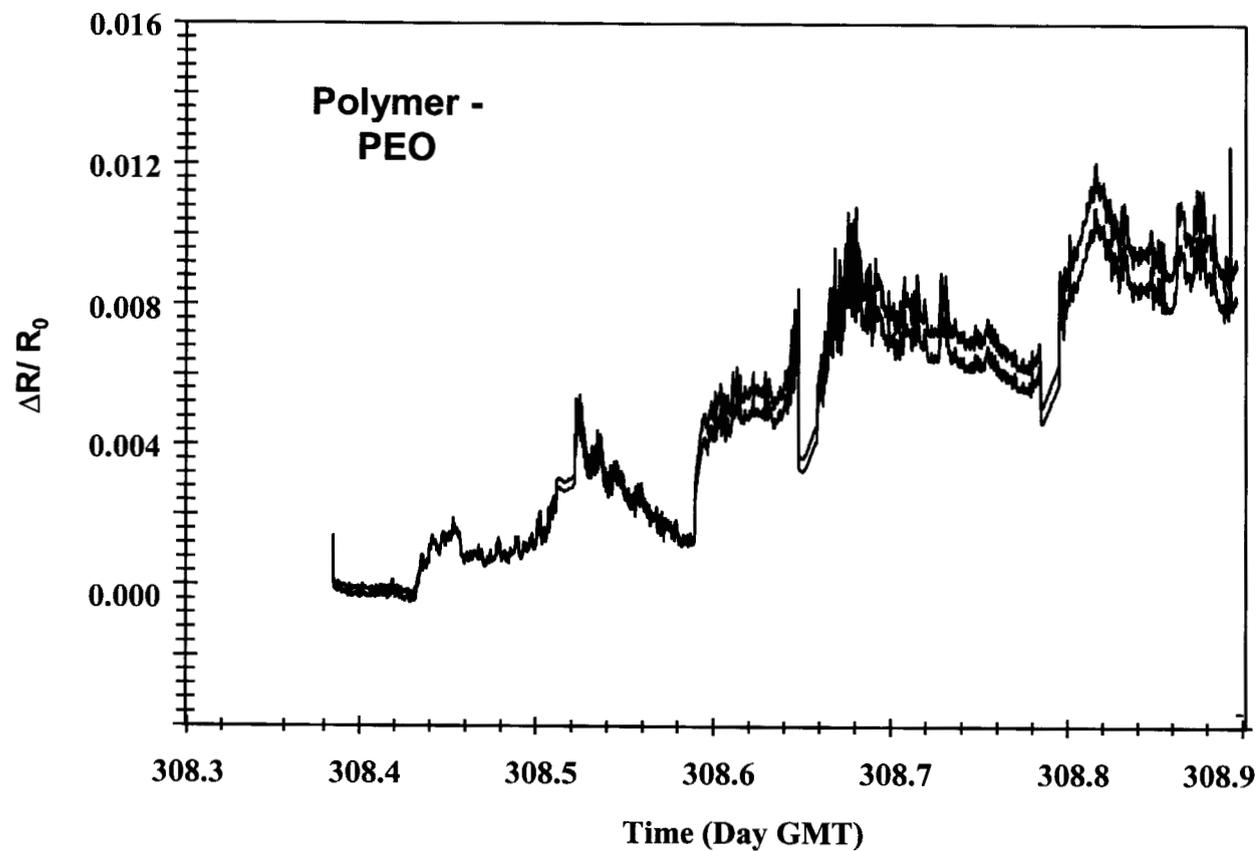
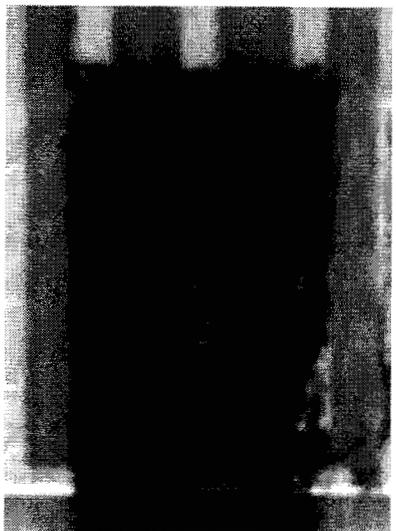
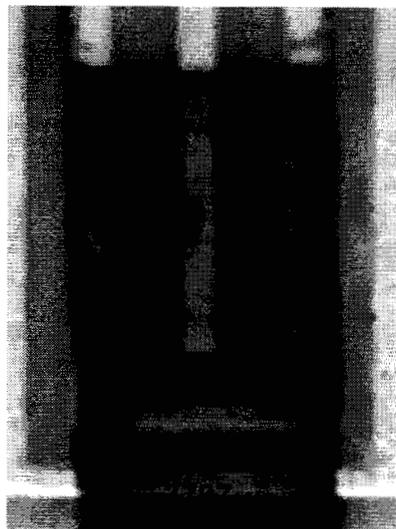
**◆ ARRAY - *TRAINING SET LIFETIME***

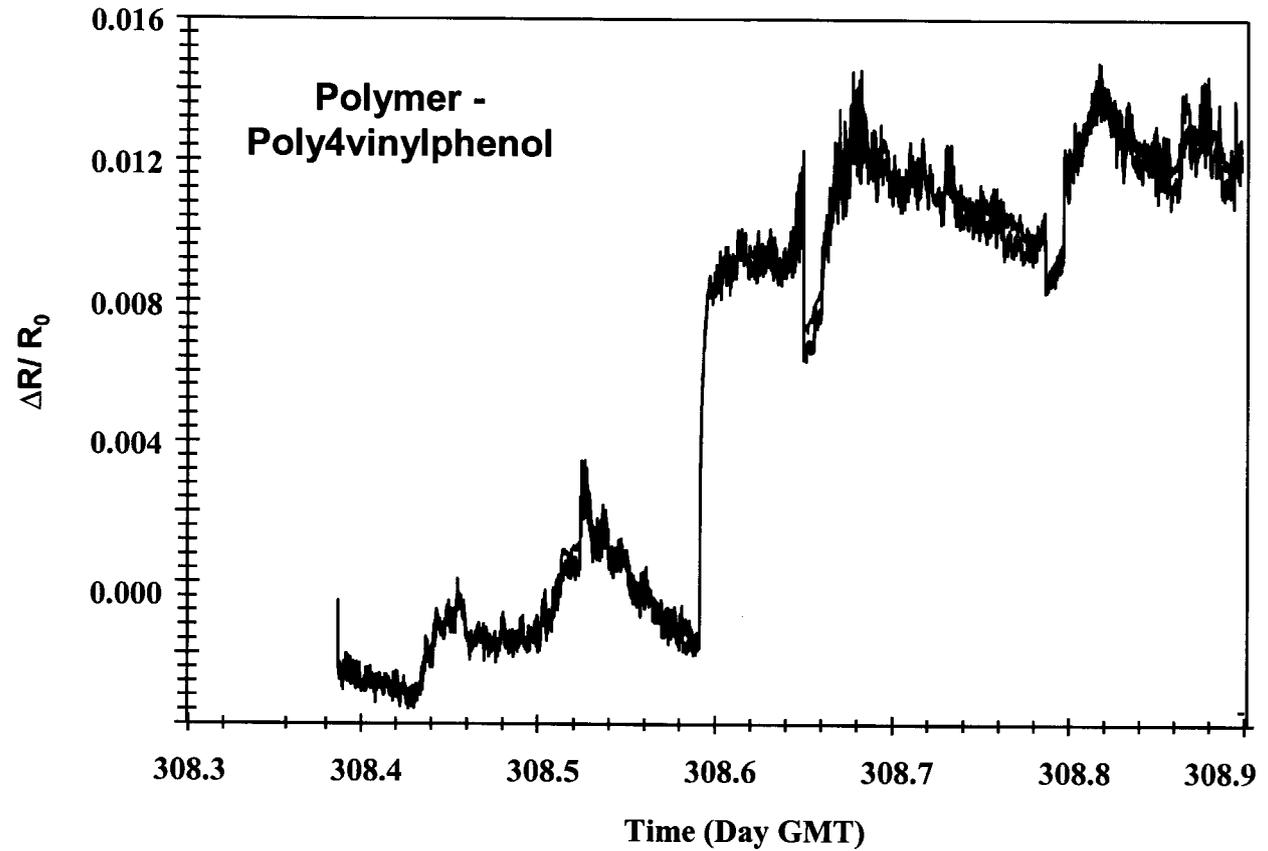
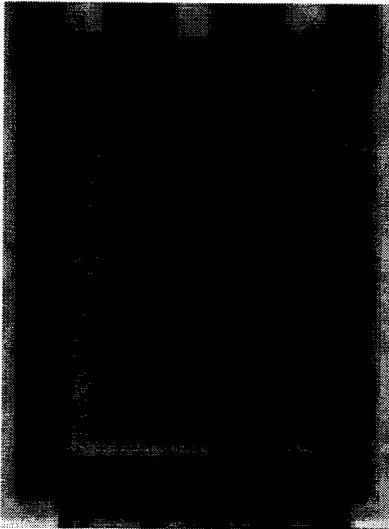
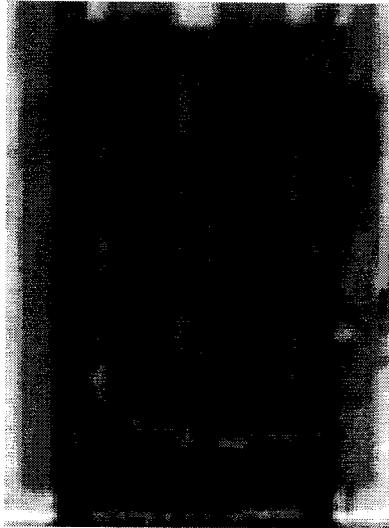
- ❖ A single training set can be used to identify and quantify analytes
- ❖ As resistance of sensors change with time, array patterns change  
- lifetime of set

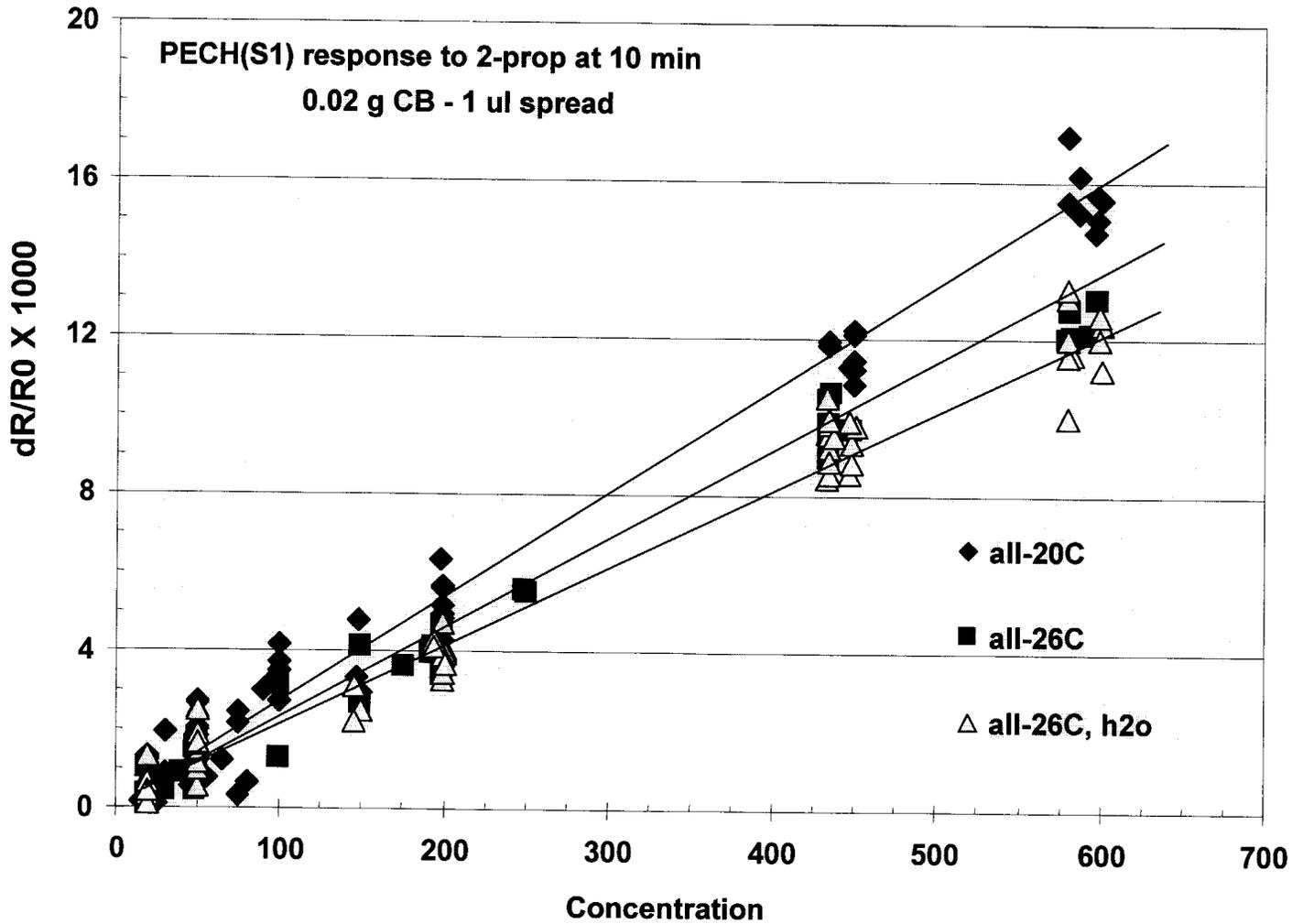


- 1 Poly(2, 4, 6-tribromostyrene)
- 2 Poly(4-vinylphenol)
- 3 Poly(ethylene oxide)
- 4 Polyamide resin
- 5 Cellulose triacetate
- 6 Poly(2-hydroxyethyl methacrylate)
- 7 Vinyl alcohol/vinyl butyral, 20/80
- 8 Poly(caprolactone)
- 9 Poly(vinylchloride-co-vinyl acetate)
- 10 Poly(vinyl chloride/acetate) 90/10
- 11 Poly(vinyl acetate)
- 12 Poly(N -vinylpyrrolidone)
- 13 Styrene/isoprene, 14/86 ABA
- 14 Poly(vinyl stearate)
- 15 Methyl vinyl ether/ maleic acid 50/50
- 16 Hydroxypropyl methyl cellulose, 10/30

Similar compounds can be distinguished by their fingerprints. Benzene and toluene are both aromatic, and have similar but distinguishable response patterns. Compounds can be classified by functional group by identifying sub-patterns in responses.

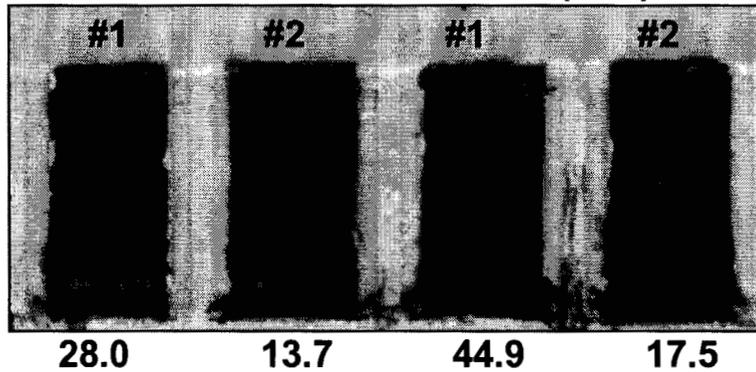




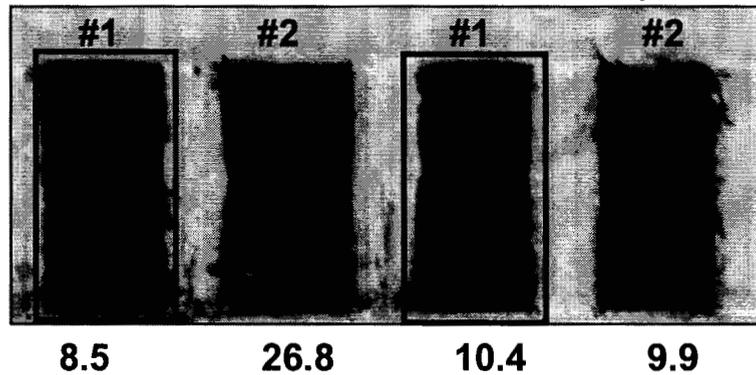


**Baseline Resistance**  
**R3 Poly 4-Vinyl Pyridine**

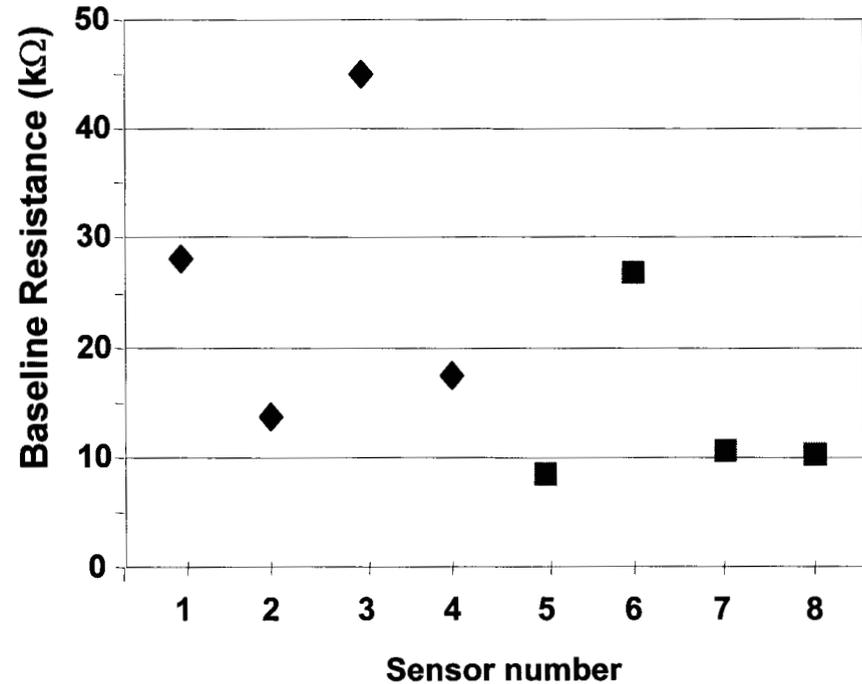
◆ Solution A 16.7% Carbon (w/w)



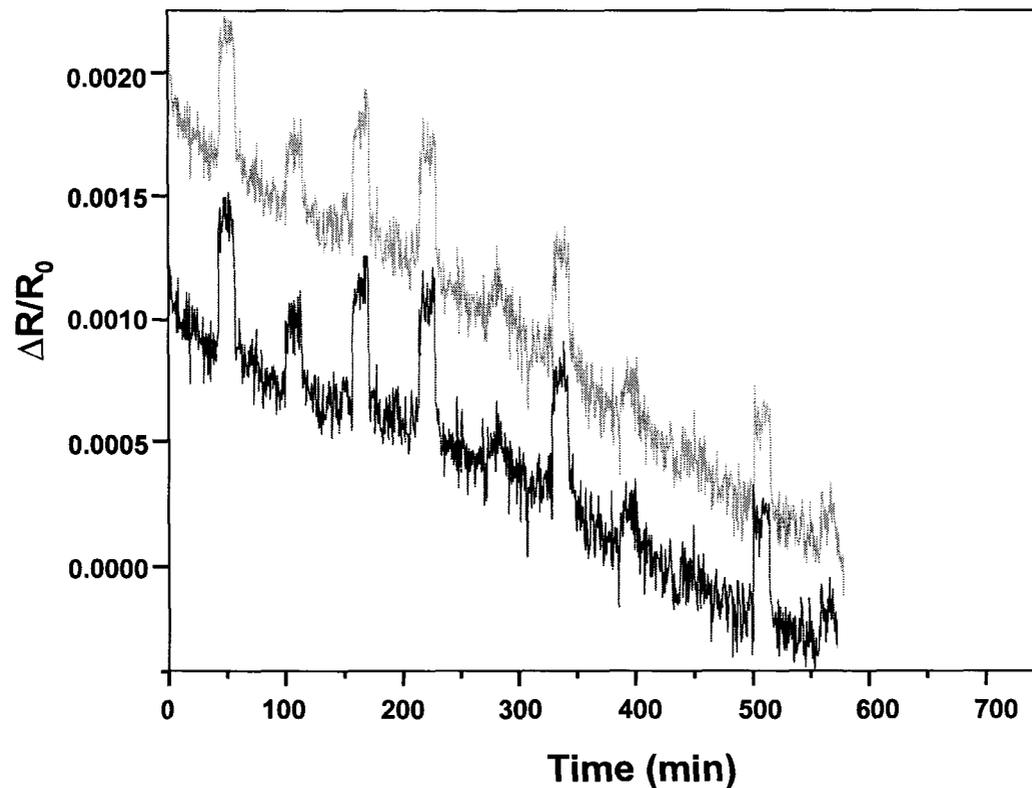
■ Solution B 16.2% Carbon w/w)



Two operators: # 1 - sensors 1,3,5,7,  
 # 2 - sensors 2,4,6,8



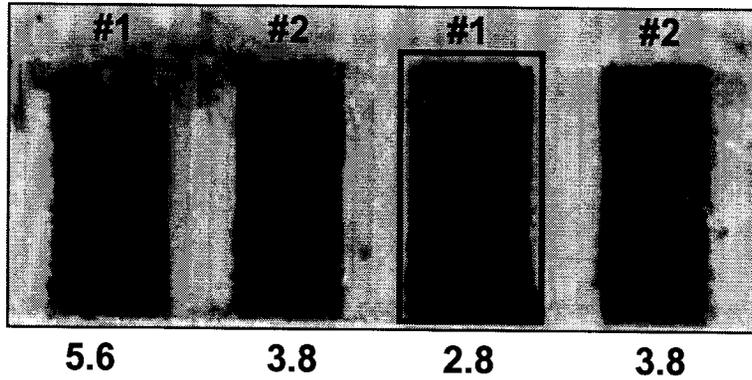
***Poly 4-Vinylpyridine***  
***Response to Hexane, 50 -1100 ppm***



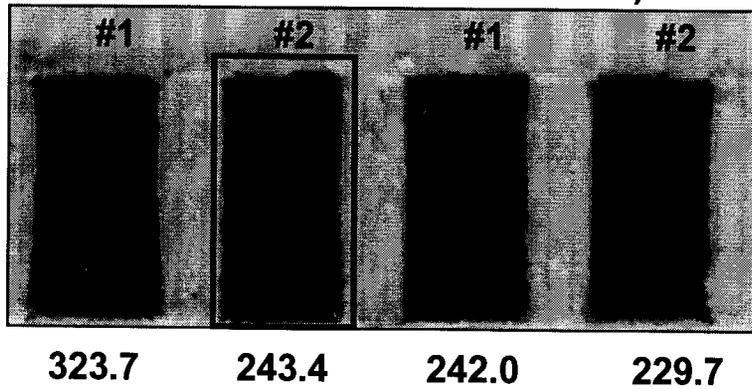
**Two sensors made from the same solution and deposited by different people at different times have identical response to hexane (sensor substrate was damaged - no data for sensors from solution A)**

**Baseline Resistance**  
**R5 Ethyl Cellulose**

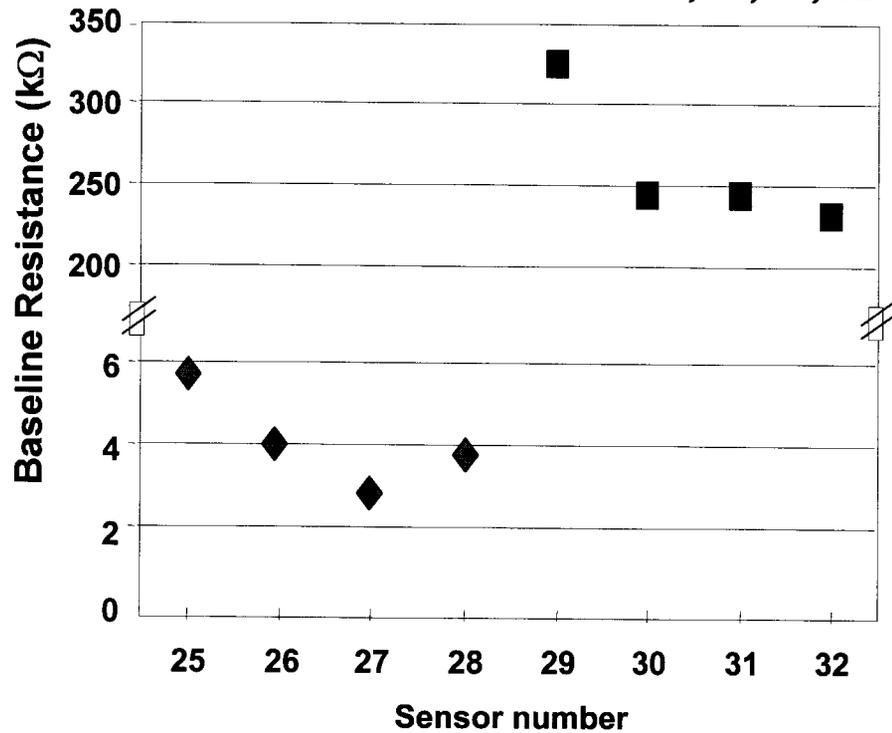
◆ **Solution A 17.6% Carbon (w/w)**



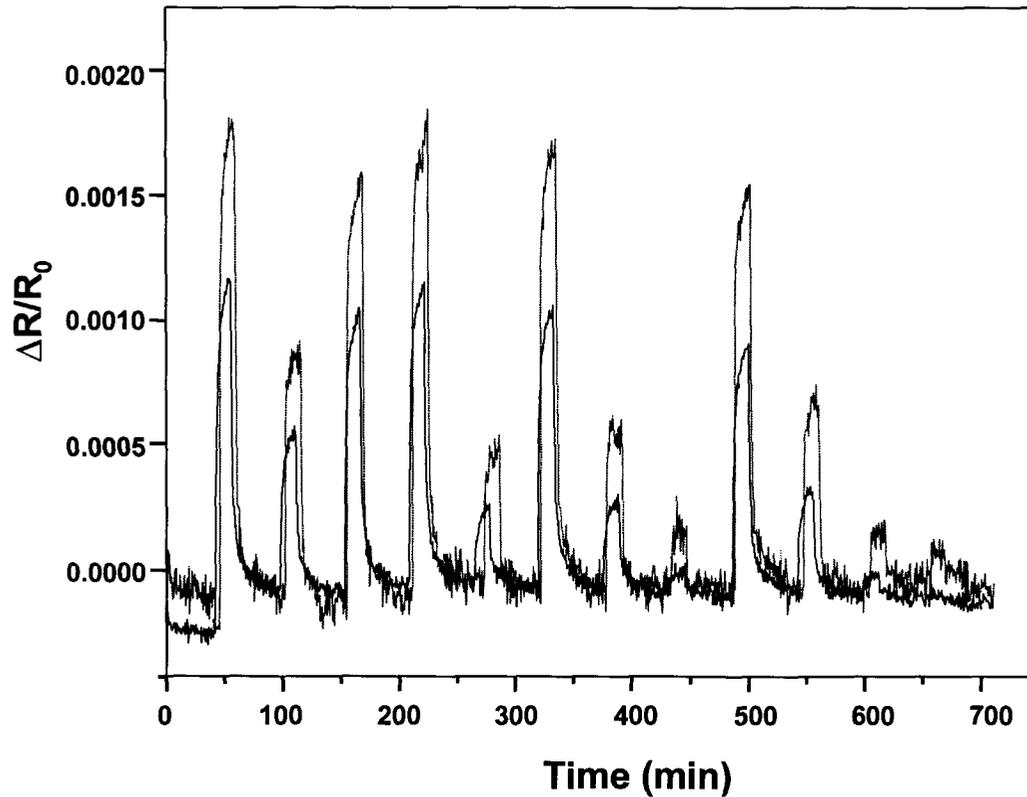
■ **Solution B 8.6% Carbon w/w)**



*Two operators: # 1 - sensors 25, 26, 27, 28*  
*# 2 - sensors 29, 30, 31, 32*



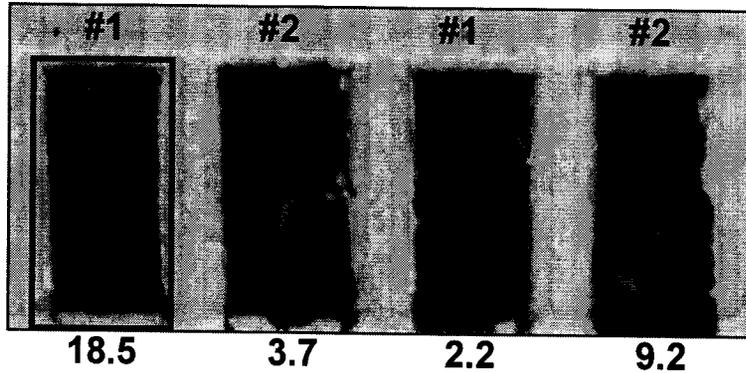
**Ethyl Cellulose**  
**Response to Hexane, 50 -1100 ppm**



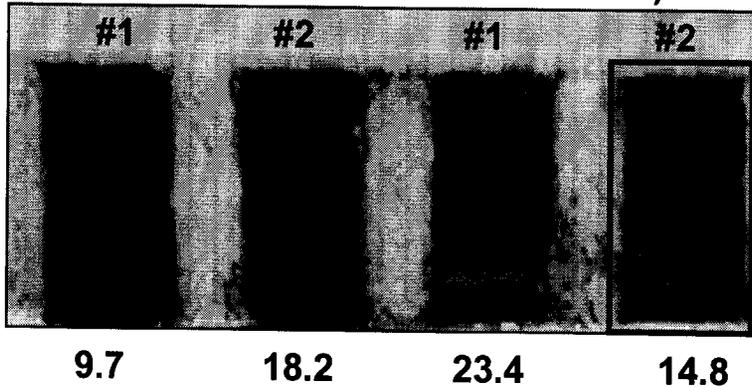
**Two sensors made from different solutions and with different carbon load have different magnitude response to hexane; lower carbon content has stronger response**

**Baseline Resistance**  
**R4 Polyepichlorhydrin**

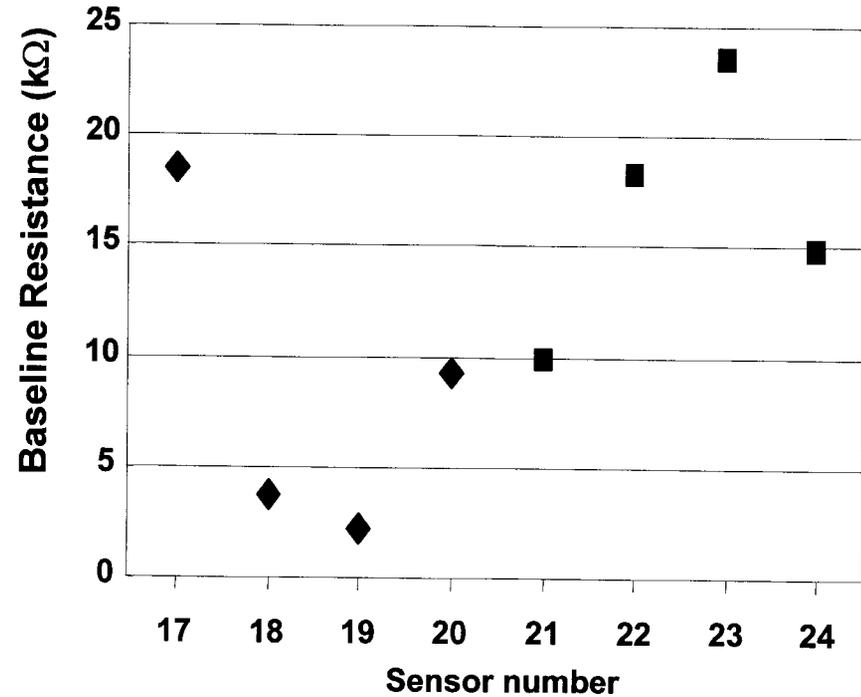
◆ **Solution A 12.1% Carbon (w/w)**



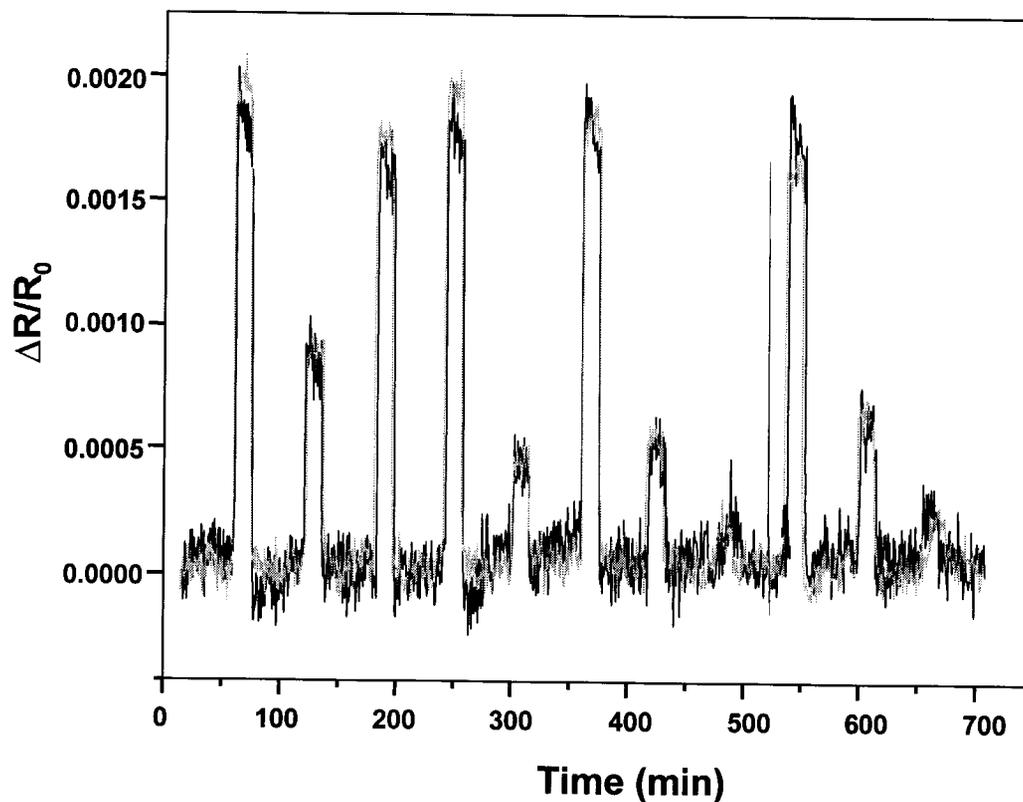
■ **Solution B 12.5% Carbon w/w)**



*Two operators: # 1 - sensors 17, 19, 21, 23*  
*# 2 - sensors 18, 20, 22, 24*

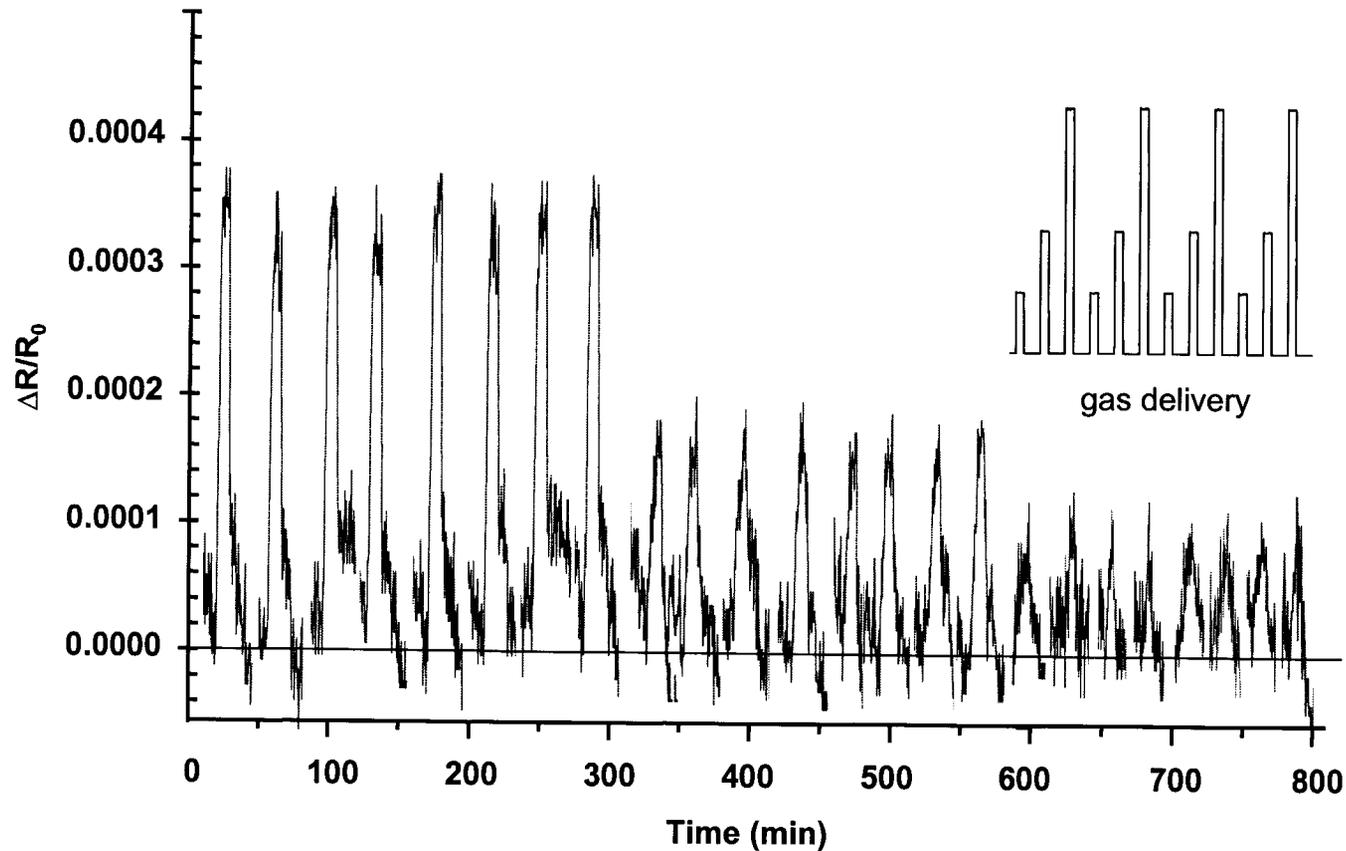


*Polyepichlorhydrin*  
**Response to Hexane, 50 -1100 ppm**



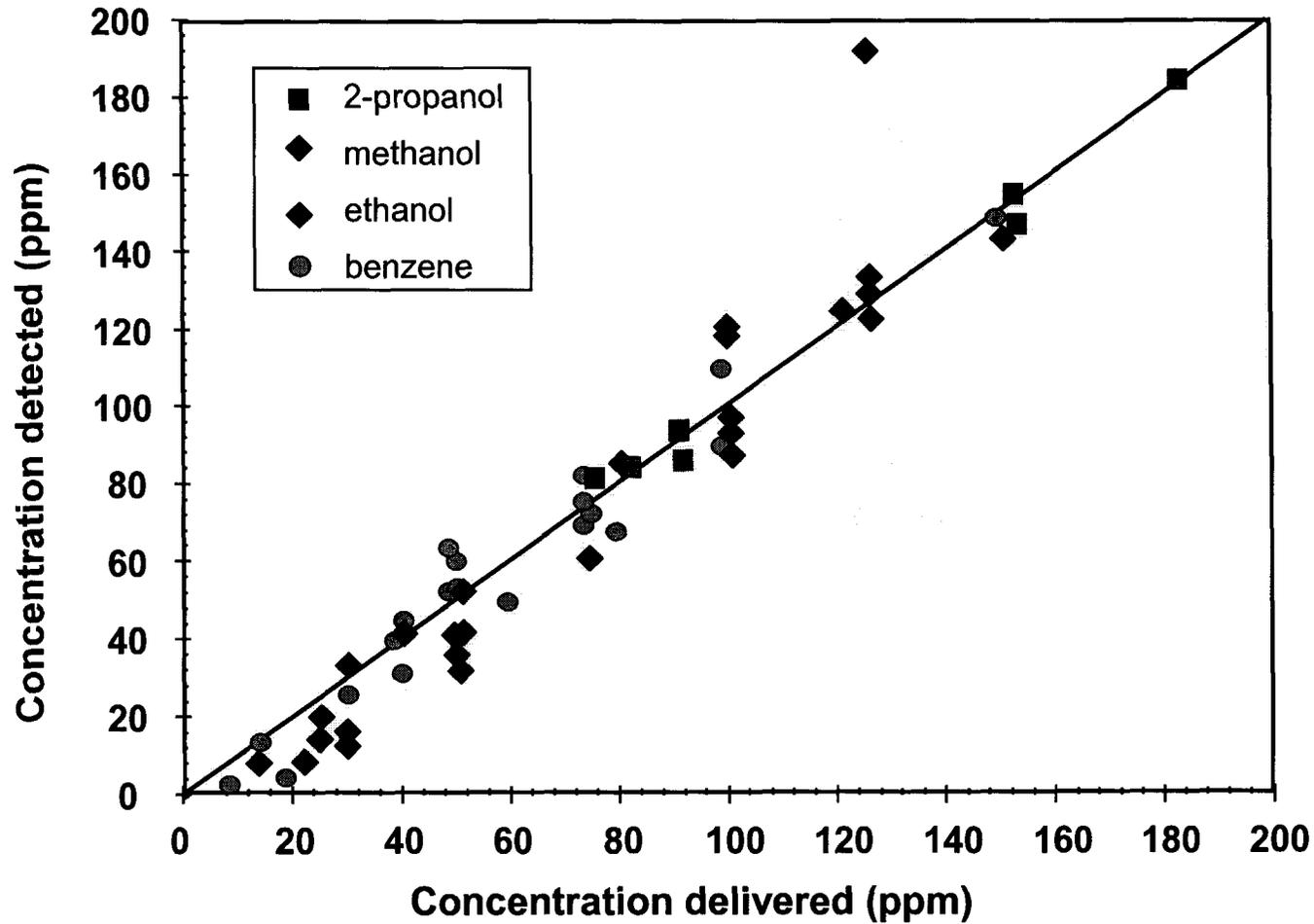
**Two sensors made from different solutions and deposited by different people have identical response to hexane**

***Polyepichlorhydrin***  
***Response to Hexane, 30 - 60 ppm***



**Two sensors made from different solutions and deposited by different people have identical response to hexane**

Identification and Quantification of Repeated Stimulus by Single Analytes



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- ◆ **Reproducibility of Baseline Resistance and Noise**
    - ❖ **Sensors made by same or different person from same solution do not always have the same baseline resistance**
      - ❖ **within same order of magnitude**
      - ❖ **appearance of films is similar**
    - ❖ **Sensors of a single polymer have similar levels of noise**
  
  - ◆ **Reproducibility of Sensor Response**
    - ❖ **Sensors made from the same solution by same or different person have similar response to analyte**
    - ❖ **Sensors made from different solutions by the same or different person have similar response to analyte**
    - ❖ **Sensor response ( $\Delta R/R_0$ ) is independent of baseline resistance if the resistances are of the same order of magnitude**
    - ❖ **Sensor response ( $\Delta R/R_0$ ) tends to increase with increasing resistance (orders of magnitude)**

- 
- ◆ **Repeatability of Sensor Response**
    - ❖ **Sensor response to the same stimulus is repeatable**
  
  - ◆ **Repeatability of Array Response**
    - ❖ **Repeatable array response is required to have an identification and quantification program which does not require re-calibration for every use**
  
    - ❖ **Array response is repeatable up to 18 months**



*Electronic Nose*  
**ACKNOWLEDGEMENTS**

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