Ground Testing of the JPL Electronic Nose: Mechanisms of Response in Conductometric Polymer-Carbon Composite Sensors

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AIR QUALITY MONITORING REQUIREMENTS

Monitor cabin environment with time stamped measurements and rates of change

- Major constituents - "near-continuous" monitoring
  N_2 O_2 CO_2 H_2O
  H_2 CH_4 CO
- Trace contaminants - less frequent monitoring required
  ~ 40 organic compounds
- Event Monitoring - rapid response time
  - pyrolysis markers - CO HCN HCl
  - marker compounds for electronics overheating
  - monitoring for sudden release from fluid systems, experiments, EVA, waste
  - follow progress of decontamination after an event

based on AEMC *Requirements for Technology Development*, 1998, Section 5

AIR QUALITY MONITORING

JPL ELECTRONIC NOSE

FUNCTIONS

- Incident monitor for targeted contaminants exceeding Spacecraft Max Allowable Concentration (SMAC). Identify and quantify target compounds at SMAC level.
- Monitor for presence of compounds associated with fires or overheating electronics
- Monitor clean-up process

CHARACTERISTICS

- Low mass, low power device
  First Generation 1.5 kg; ~ 2 L; 1.5 W avg, 3 W peak
  Second Generation < 1 kg; < 1 L; 1.5 W avg, 3 W peak
- Requires little crew time for maintenance and calibration

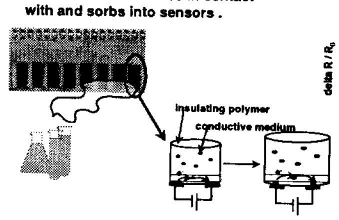
WHAT IS AN ELECTRONIC NOSE?

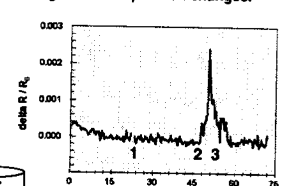
An array of non-specific chemical sensors, controlled and analyzed electronically, which have overlapping responses to compounds. Compounds are identified and quantified by recognition of patterns of response.

1. ENose measures background resistance in each sensor and establishes R_0 (baseline).

2. Contaminant comes in contact with and sorbs into sensors.

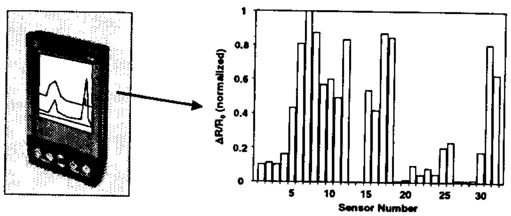
3. The sensors, polymer films loaded with a conductive medium such as carbon black, change resistance by swelling or shrinking as air composition changes.





WHAT IS AN ELECTRONIC NOSE?

4. Resistance is recorded, the change in resistance is computed, and the distributed response pattern ("fingerprint") of the sensor array is used to identify gases and mixtures of gases. The magnitude of response is used to quantify the identified compound.

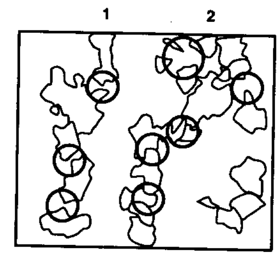


5. Responses of the sensor array are analyzed and quantified using software developed for the task.

n-propanol, 30 ppm

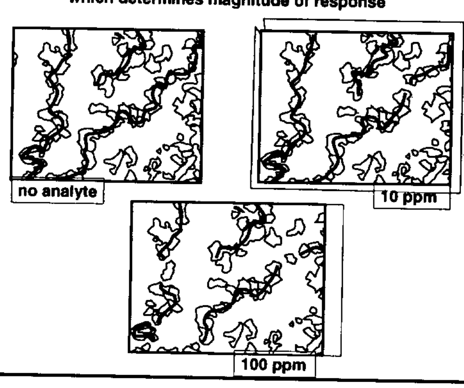
CONDUCTION IN POLYMER-CARBON COMPOSITES

1. **Tunneling**
   - Carbon to carbon *through polymer matrix*
   - Resistance is controlled by electric field between adjacent particles
2. **Percolation Controlled**
   - Carbon-to-carbon through *particle contact*
   - Resistance is controlled by contact resistance between particles and by *gaps too large for tunneling*



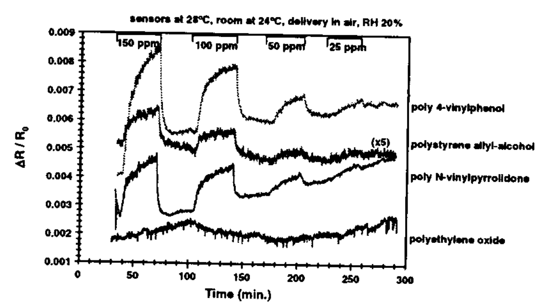
CONDUCTION IN POLYMER-CARBON COMPOSITES

Concentration of analyte determines degree of swelling, which determines magnitude of response



CONDUCTION IN POLYMER-CARBON COMPOSITES

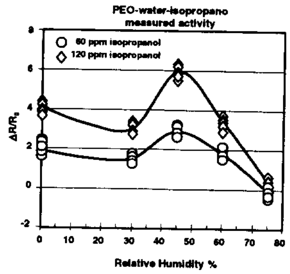
Resistance Change: Response to Methanol

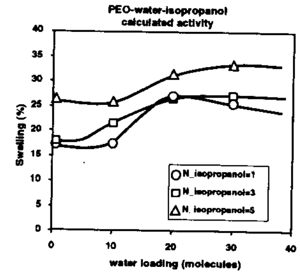


CONDUCTION IN POLYMER-CARBON COMPOSITES

Resistance Change: Response to i-propanol

dip in response magnitude at increased humidity shown in calculated swelling behavior

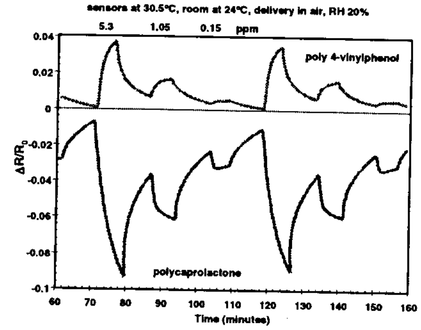




CONDUCTION IN POLYMER-CARBON COMPOSITES

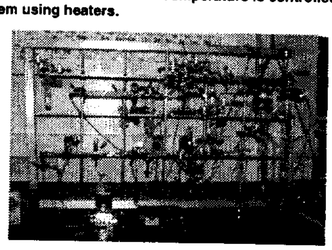
Resistance Change: Response to Hydrazine

sensors at 30.5°C, room at 24°C, delivery in air, RH 20%



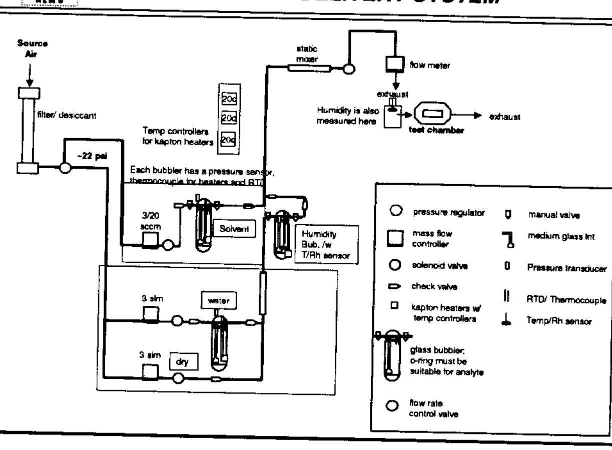
AIR / ANALYTE DELIVERY SYSTEM

A computer-controlled gas delivery system bubbles air through solvents and mixes them with humidified air. Temperature is controlled in the gas delivery system using heaters.



- concentrations are verified using a Rosemont Hydrocarbon Analyzer calibrated to standard gases (methanol, methane, toluene, acetone). Humidity is verified using calibrated sensors.
- Background gases can be cleaned air or bottled gas. Humidity is added by bubbling through water and mixing humidified air with dry air.

AIR / ANALYTE DELIVERY SYSTEM



100 LITER TEST CHAMBER

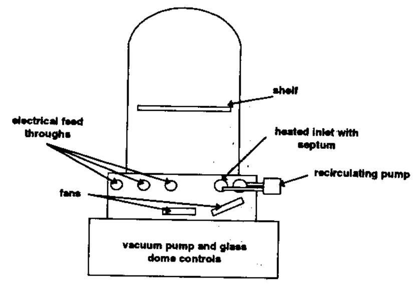
DEVICE OPERATION AND TESTING

Test Chamber

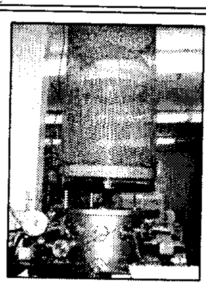
- 100 L test chamber
- Temperature controlled using a water jacket
- Pressure controlled by evacuating and backfilling with clean, humidified air
- Test analytes are delivered using a syringe for rapid injection (to simulate a spill) or a syringe pump (to simulate a leak).
- Solid analytes (indole) are delivered by placing a measured quantity in a feedthrough. Gaseous analytes (burning insulation) is delivered through a tube.
- Chamber air can be circulated within the chamber or directed through a filter to simulate the Air Revitalization System.

- Testing will begin after a complete training set has been taken. Data analysis is automated to find events and identify them.

100 LITER TEST CHAMBER



100 LITER TEST CHAMBER

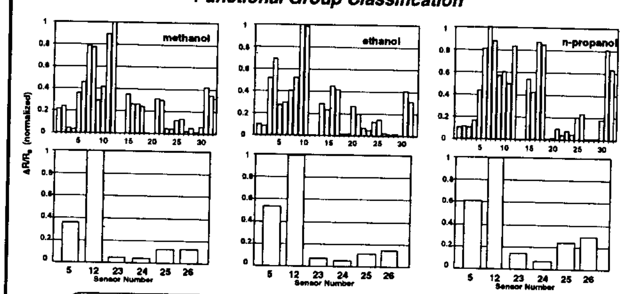


Temperature and Pressure Controlled 100 L Test Chamber

- Liquid test analytes are delivered using a syringe or a syringe pump
- Solid analytes are delivered by placing a measured quantity in a feedthrough; gaseous analytes are delivered through a tube.
- Chamber air can be circulated within the chamber or directed through a filter to simulate the Air Revitalization System.

DATA ANALYSIS

Functional Group Classification



Three primary alcohols (methanol, ethanol, 1-propanol) can be distinguished by their response patterns. A sub-pattern made up of five sensors has been identified as being indicative of primary alcohols. This technique can be used to identify compounds by functional group.

DATA ANALYSIS

Functional Group Classification

Classify Unknowns In Mixtures and/or With More Than One Functional Group

preliminary software was for single unknowns, with a single functional group. focus was on alcohols and aromatics.

developed software routine for choosing best subsets of sensors for functional group classification based on fingerprint group distance calculation. Software will find a subset of sensors that has a minimum value of the distance between one particular group divided by the distance between the rest of the target analytes. Each different functional group has a different optimal subset of sensors, and so do different combinations of functional groups. This sensor selection software is a very useful tool and may be applicable to other general sensor selection /evaluation needs.

Also devised a two-step data analysis approach in which the result of the first (main) step will be given preference in the second (functional group analysis) step. For example, if the first step results in "methanol" plus "freon" with an unacceptably large residual (r_0), then the software will assume it could be of some unknown analytes.

DATA ANALYSIS

Functional Group Classification

trained analytes	untrained
methanol	2-propanol
ethanol	butanol
1-propanol	pentanol
methane	phenol
ammonia	anthracene
benzene	ethyl benzene
formaldehyde	p-xylene
freon113	o-xylene
indole	m-xylene
toluene),	

mixture events were generated (simulated) from one untrained analyte plus one trained or untrained analytes

Total tested are 171 different combinations of binary mixtures; successis correct functional group classification

success rates	
2-propanol	86%
butanol	67%
pentanol	76%
phenol	67%
anthracene	86%
ethyl benzene	95%
p-xylene	95%
o-xylene	86%
m-xylene	100%

Analytes for 2nd Generation Enose

	24 hr SMAC	1/3 SMAC	3x SMAC	tested limits low	tested limits high
acetaldehyde (1500ppm gas)	6	2.00	18	2	20
acetone	200	66.67	600	65	600
acetonitrile (1500 ppm gas)	4	1.33	12	1	25
ammonia - NH3	20	6.67	60	6	60
benzene (1510 ppm gas)	3	1.00	9	3	75
2-butanone	50	16.67	150	15	150
chlorobenzene	10	3.33	30	3	30
dichloromethane	35	11.67	105	11	150
dichloroethane	0.4	0.13	1.2	TBD	TBD
ethanol	2000	666.67	6000	665	6000
ethyl benzene	60	20.00	180	18	165
formaldehyde	0.1	0.03	0.3	TBD	TBD
freon 113	50	16.67	150	15	150
furan (500 ppm gas)	0.36	0.12	1.08	TBD	TBD
hexane	50	16.67	150	16	150
hexamethyltricyclosilane	25	8.33	75	TBD	TBD
hydrazine	0.3	0.10	0.9	TBD	TBD
indole	0.3	0.10	0.9	TBD	TBD
methane	5300	1766.67	15900	3000	40000
methanol	10	3.33	30	3	100
methyl hydrazine	0.002	0.0007	0.006	TBD	TBD
iso-propanol	100	33.33	300	30	600
tetrahydrofuran	40	13.33	120	13	120
toluene	16	5.33	48	5	50
1,1,1-trichloroethane	11	3.67	33	5	200
o,p-xylenes *(use m value)*	100	33.33	300	33	300

ACKNOWLEDGEMENTS

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