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Supplementary information

Fabricating and printing chemiresistors based on monolayer-capped metal nanoparticles

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SUPPLEMENTARY INFORMATION

Fabrication and Printing Chemiresistors Based on Monolayer-Capped Metal Nanoparticles

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Supplementary Data 1 | Masks design

The scheme of the sensor electrodes shows on Supplementary Fig. 1. Each device includes eight sensors with the diameter 1 mm. The electrodes width is 10 microns and gap of 10 microns. The external size of the device is 150 mm x 153 mm, in addition the device includes two resistors; the first resistor is used for the temperature control and the second one is for temperature measurement. The fabrication process consists of only two photolithography steps. The first is for metal electrodes deposition and the second one is for polymer micro-barrier creation.

Supplementary Data 2 | The sensors response examination

The sensors response examination is performed by the system consist of special sample chamber, permeation oven that connected with nitrogen gas tubing and multimeter as shown in Supplementary Fig. 3. A computer with specific software controls the measurement system (see Supplementary Manual 2)

The sensors layout

The PCB board is designed to measure two devices. Each device contains eight individual sensors and two resistors for temperature control.

Sensor Printed Circuit Board (PCB) layout

In order the examination, the sensors are inserted to the PCB. The board was produced in Cellix Ltd. Longmile Business Centre, Longmile Road. Dublin. Each board includes two chip holdings with spring contacts, connector, humidity and temperature sensors, capacitors and resistor (Figure 2)

Supplementary Data 3 | Synthesis of Dodecanethiol caped gold nanoparticles

! CAUTION The synthesis performed under hood.

1. Turn on the digital hot-plate stirrer to 800 rpm and adjust the temperature to 30°C. Place a water bath on the heater and wait until the temperature has stabilized. The round-bottom flask will be kept under these conditions throughout the synthesis.
2. Gold salt solution preparation. Dissolve 310mg of $HAuCl_4 \bullet xH_2O$ in 25ml of DDW (31.5mM). ▲ **CRITICAL STEP** The DDW must be fresh from the same day of synthesis. $HAuCl_4 \bullet xH_2O$ should be transformed to a glass vial using a plastic or Teflon spatula. 25 ml of DDW should be measured using volume measuring funnel. Mix the $HAuCl_4 \bullet xH_2O$ in DDW by shaking the vial. Color of the solution is transparent yellow.
3. Tetraoctylammonium bromide (TOAB) solution preparation. Dissolve 1.5g of TOAB in 80ml of toluene (34.3 mM). Toluene is measured with volume measuring funnel. Then transferred to a round-bottom flask of 500ml volume. TOAB is measured on weighting paper and transferred directly into the toluene solvent in the round-bottom flask. Add a stirring bar. This mixture is stirred for 10 mins at RT and 800 rpm. Color of the solution is transparent.
4. Transfer of gold salt to toluene. Transfer $AuCl_4$ [step 2] from aqueous $HAuCl_4 \bullet xH_2O$ solution (25ml, 31.5mM, 310 mg) to [step 3] a toluene solution by the phase-transfer reagent TOAB (80ml, 34.3mM, 1.5g). Mix both solutions in a separating funnel. After separation, organic phase is on the top having brownish color. Aquas phase on the bottom having transparent color. Then isolate the organic phase in separating funnel.
5. Transfer the content of $HAuCl_4$ with TOAB in toluene into a round bottom flask (at 30°C) and stir it vigorously at 800 rpm via magnetic stirring plate and stirrer. Then add 219 µl dodecanethiol using a digital pipette by fast injection. ! CAUTION Handle the dodecanethiol in a chemical hood. Keep the pipette and the gloves in the hood for a few hours (See Supplementary Fig. 5a).
6. Reducing agent preparation: The 25ml of DDW is measured with volume measuring funnel. Transfer DDW to a vial and cool it with ice for 10 min. Then dissolve 380 mg (in a glass vial) of $NaBH_4$ (0.4M) using the cooled water. Mix the solution by shaking. Use this solution immediately after mixing.
7. Nucleation and growth of gold NPs add ice-cold $NaBH_4$ solution from step 6 into the $HAuCl_4$-TOAB in toluene solution [step 5] at 4°C 800 rpm on magnetic stirring plate using magnetic stirrer. ! CAUTION $NaBH_4$ is a very strong reducing agent.
8. The reaction is allowed to occur under stirring at 800 rpm at 4°C for at least 3h. ▲ **CRITICAL STEP** It is important to maintain same temperature and stirring rate during the synthesis.
9. A purification step of GNP starts with separating the water and organic phases by using the separating funnel. Organic phase is the upper dark brown phase. The lower transparent phase is the water. (See Supplementary Fig. 5b)
10. Drain the water phase and discard it.
11. Transfer the organic phase from the separating funnel (which contains the gold NPs) to a round flask 500ml
12. Connect the flask to a rotary evaporator according the instrument instructions (bath at 40°C, rotating speed 140-170 rpm). Usually, it takes about 15-20 min to evaporate all 80ml of toluene. At the end of the process, there should be no solvent and only NPs residuals. (See Supplementary Fig. 5c)
13. Separate the flask from of the evaporator according the instrument instructions.

14. Insert 5ml of toluene to the flask.
15. Dissolve all GNPs in the toluene.
16. Add 400ml ethanol and store it for overnight (18-20h) in the freezer (-10°C).
    ? TROUBLESHOOTING
17. Transfer all the liquid content into centrifuge bottles. Centrifuge for 10 min at 6000 rpm. Note: At the end, you should have a NPs pellet stuck to the walls of the bottles.
18. Discard the solvent. After the NPs are dry on the walls of the bottles, dissolves them again in toluene.
19. Take small round flask (25ml), weigh it, and record its weight. Write it as A gram.
20. Put the NPs solution in toluene.
21. Use the rotary evaporator again as described above.
22. Weigh the flask again when the solvent is evaporated. Write it as B gram.
23. Calculate how much solvent you need to add in order to have the desired concentration of toluene to add ((B-A)*1,000)/20). After dissolving, transfer the solution into a new glass vial and seal with parafilm. ▲ **CRITICAL STEP** Store all NPs solutions at 4-6°C in glass vials.
24. Inspect the GNPs solutions by TEM to examine the shape, diameter, size distribution of the GNPs, and purity of the GNPs. (Fig. 3), e.g., shows GNPs with organic modification of dodecanethiol and GNPs with 2-ethylhexanethiol modification.

Preparation of MCGNPs concentrations:

25. Take small round flask (25ml).
26. Weigh it and record its weight = A gram.
27. Put the NPs solution in toluene.
28. Use the rotary evaporator again as described above.
29. When the solvent is evaporated weigh the flask again = B gram.
30. Calculate how much solvent you need to add in order to have the desired concentration= ((B-A)*1000)/5 => Cml of toluene to add. After dissolving, transfer the solution into a new glass vial and seal with parafilm.

All MCGNPs solutions are stored at 4ºC in glass vials up to two months.

Supplementary Data 4 | Effect of different micro-barrier modification on sensors response

Few types of micro-barrier material were evaluated: SU-8 and silicon oxide. Supplementary Figure 6a shows a droplet of 20nl of GNPs modified with 2-ethylhexanthiol resulted non-uniform film that run over the external boarder of the electrode. The addition of a micro-barrier made of SiO_2 (Supplementary Fig. 6b) restricted the flow of the printed emulsion in an efficient manner, though the sensors still adopt different morphological shapes. Conversely, when the barrier was made of SU-8, the devices were held in its place and adopted a uniform structure. Functionally, when the uniformity of response of these sensors were compared upon exposure to different doses of octane, it was found that the addition of the micro-barrier made of SU-8 helps to unify the sensor response as well. Indeed, the variability indexes (VI) changed from 4.4% in the case of the bare electrodes, to 3.4% in the case of SiO_2 micro-barrier, to 2.4% in the case of the micro-barrier made of SU-8.

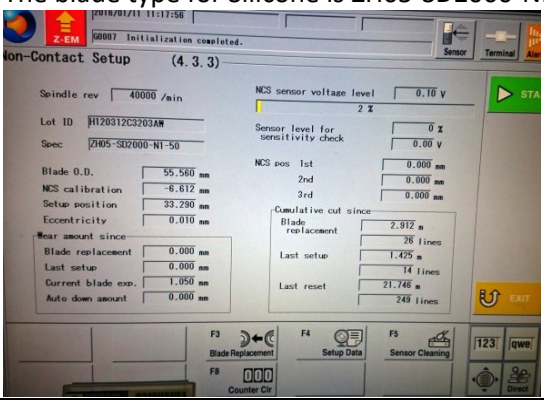
To verify that the aforementioned observations are not a unique for a specific organic ligand, we choose to functionalize the GNPs with an additional functional group. 20 nl droplets containing GNP modified with dodecanethiol were printed on the electrodes. The observations were similar to those obtained with GNPs modified withe 2-ethylhexanethiol. As seen in Supplementary Figure 7a, the droplet was not uniform and was flowing outside the electrode. Addition of a micro-barrier made of SiO_2 (Supplementary Fig. 7b) restricted the flow of the printed emulsion in an efficient manner. Conversely, when the barrier was made of SU8, the casted sensors were held in their place and adopted a more uniform structure than in the case of micro-barriers made of SiO_2. The VI value changed from 15.2% to 9.2% in the case of the micro-barrier made of SU8 (Supplementary Figure 7d, e & f). In the functional realm, it was shown that the addition of the SU-8-made micro-barrier helped to achieved more uniform sensing responses.

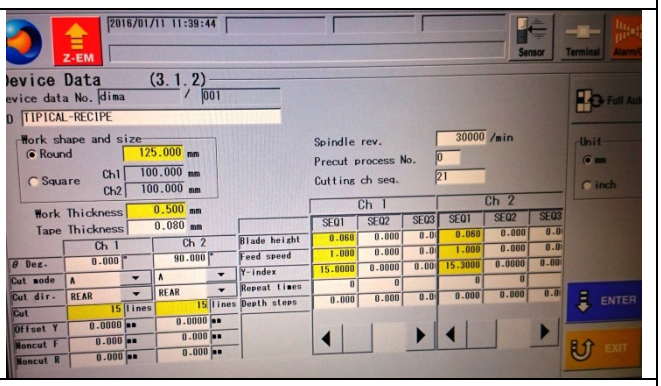
Supplementary Manuals

Supplementary Manual 1 | Dicing operation

In order to cut the 4 inch wafer consist of 21 devices use dicer machine DISCO DAD 3350. Table 1 shows the step-by-step instruction for the Si wafer cutting.

Table 1: Dicing operation

#	**Process**	**Remarks**
1	Turn on following: 1. Water line 2. Compressed air line 3. The DISCO machine	The machine operation mast be with water and compressed air. The water flow is 0.6 L/min 
2	Turn on System initialization and wait to be complete it.	
3	Frame Preparation Frame entering	Clean the frame and the back side of the wafer with Isopropyl alcohol (IPA). Attach the dicing tape on the frame. Place the wafer in the middle of the frame. Flip the frame with wafer with the top on clean paper and press smooth out the tape. Load the frame to the holder. Turn on the C/T Vacuum
4	Blade setup -> Start When the setup is completed push Enter Check a type of blade – if you need change the blade do blade replacement. Exit	The blade type for Silicone is ZH05-SD2000-N1-50 

5	Check the Spindle. It must be turn on.	Spindle speed is 30000 in min
6	**Data definition:** Go to Device data –> Typical recipe -> Enter Enter the parameters: Work size 125mm, Work thickness 0.5mm (the wafer thickness), Y-index: Ch1= 15.05 mm, Ch2= 15.35mm Feed speed is 1 mm/sec Exit	
7 7.1 7.2 7.3	**Dicing process:** Go to Manual operation -> Cutting SemiAuto -> Manual Aligment -> Change magnification Do alignment of the wafer from one edge to another. Enter -> Exit Check the number of lines for cutting. (6 lines in each channel). Press Start for cutting Double Start for pause and checking the cut line. If a cutting line not on the middle -> Go to the middle of required line -> Do Adjustment Turn the wafer 90 degrees. Check the number of lines for cutting. Push to wafer button and go up or down by arrow buttons. Press Start bottom.	Start the alignment from the first row on the wafer. For the more accurate alignment go to high-resolution magnification. For the checking push the wafer button and go up or down by arrow buttons. 
8	End of work: Exit-> Exit ->C/T vacuum Turn off the machine. Turn off the water and air flow.	Take out the frame with the wafer and carefully cut the tape surround the wafer.

Supplementary Manual 2| Scienion sciFLEXARRAYER S3 dispenser operation

This user manual describes operating the sciFLEXARRAYER S3 system for printing the different types of MCNPs. Scienion sciFLEXARRAYER S3 (Supplementary Fig. 8) is an automated piezoelectrically-driven non-contact, drop on demand picoliter dispensing system This piezoelectric dispenser system allows printing of precise drops of 0.3±0.02 nl. The system concludes of dispensing robot, pumping unit for samples holding, humidity and dew point control system, system liquid bottle, wash supply, PC and filtered hood. The robot station contains XYZ axis stage, CCD camera, source solution holder and target holder.

! CAUTION The system use toluene as the basic solution. Keep all the system into hood.

The general user rules:

Centrifuge your samples (or filter them with a 0.22µm filter if possible) prior to aspirating them with the PDC. Don't leave your sample inside the PDC for a long period of time (i.e., longer than it is required for a run) to minimize the chance of precipitation or crystallization inside the PDC.

Clean your PDCs daily, especially before shutting down the system.

Procedure:

1. Turn on: machine PC, SciFlex_s3 program, dry air insertion, vacuum pump.
2. Go to File -> Humidity control. Verify chamber conditions are: Temp = 20±2°C, RH = 38±2%
3. Load the PDC 70. Do not connect it yet to the manifold. Load Well 384 and wafer.
4. Prime washing with water.
    - 4.1 Connect the flush bottle to the manifold.
    - 4.2 Connect the PCD to the valve after verifying drop existence.
    - 4.3 Continue the prime washing.
5. Washing with Toluene Change the system liquid bottle with water to toluene bottle.
    - 5.1 Do task ->Nozzle removal wash. Disconnect the PDC and connect the flush bottle to the manifold.
    - 5.2 Start machine washing. Robot setup -> Syringe pump. Set up a volume of 9000µl and speed 90µ/s.
    - 5.3 Disconnect the flush bottle in the end of the washing. Set up a volume of 200µl/s and speed 10µ/s then connect the PDC to the manifold when a first drop appears.
6. The main parameters definitions. Go to Main (see Supplementary Fig. 9)
    - 6.1 Choose prob 384Genetix Vshape on the probe window.
    - 6.2 Choose run program "SpotRun". This program includes the next steps: BeginLoop, SpotProbeRun, EndLoop, MoveHome
    - 6.3 Load the coordinate file On Target:
7. Nozzle parameters setup**:**
    - 7.1 Insert following parameters, written on nozzle package and/or on the enclosed certificate:
        - Voltage: 90V
        - Pulse: 50µs
        - Frequency: 300Hz
        - Led Display: 200 µs

Save the parameters by pressing the set nozzle parameters button (See Supplementary Fig. 10)

7.2 Go to the Nozzle Offset (Supplementary Fig. 10) and press on the Camera Station button

7.3 The center of the front edge of the nozzle tip has to be on the image center (green cross on Fig. 13b). Use right or left buttons to move on axis X and up or down to move.

7.4 Press on Autofocus button to determine the exact Y offset. Autofocus function helps to find the correct position and offset using a reproducible evaluation of the drop image.

7.5 Optimize the drop generation with Toluene:

7.5.1 In Dispense Mode select Continuously Dispensing, click on Start button to initiate the dispensing (Supplementary Fig. 10).

7.5.2 Modify the parameters: **voltage, pulse, frequency** to get a stable drop image at the right distance to nozzle. Typically, the drop should still be stable when varying the parameters by approx. ± 2 units (the nozzle parameters written on its package and in the enclosed certificate). The distance between drop and nozzle (at an LED delay of 200 µs) in the camera image is an indicator for the speed. This distance should be kept in the range of 450 to 500 µm. (Supplementary Fig. 10)

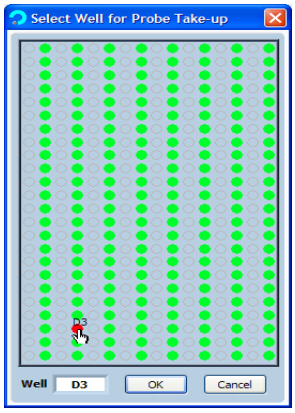
7.5.3 Once the drop is stable, press the button **Set Nozzle Parameters**.

8. Nozzles setup

Load 50µl-60µl of NP solution into selected probe type of Well384.

8.1 Go to nozzle setup

- Choose Do Task
- Choose Take Probe 15µl tasks.
- Select probe type. Press Ok.
- When syringe pump aspiration has finished, press on go home button



The well 384 mapping

8.2 Optimize the drop generation with NP solution

8.3 Go to nozzle setup

- Choose Do Task
- Press on Drop Volume to determine the volume
- Make sure you are in the drop volume recommended for PDC 70 is 300-360 pl

8.4 Calculate a number of drops [X]:

$$X = \frac{10[nl] * 10^3[pl/nl]}{Drop\ Volume\ [pl]}$$

For example, calculation for 40 nl and 300pl volume: 40000/300 = 133 drops

9. Target Setup

9.1 Go to Field Setup (Supplementary Fig. 12): insert the number of drops calculated in 8.5, Go to Pause settings and insert: pause: 0 ms after 0 drops

9.2 Select the active well by clicking on it. When the light green well can be used. When the light dark green: well has been used. Choose the field by clicking on it. When the light green appears, it means Spot position filled with sample. The table contains the entire information of the current field setup.

10. Printing

10.1 Go to Run (Supplementary Fig. 13): Check the selection of Probe, Run and Target before starting. Press start Run than Select Target List and Order will be opened automatically.

It displays the grid of targets according to the definition of the target type selected for the run. Click on each target symbol and press OK.

10.2 In order to print the next sample, make sure PDC is loaded with enough solution. If not, repeat step 8 and step 9.

11. Washing system and PDC on the end of work.
    11.1 Internal PDC70 washing Do task - Wash flash strong
    11.2 Disconnect the flush bottle and connect the PDC to the manifold, Wash PDC with toluene. Robot setup-> Syringe pump (Volume 500µl/s, Speed 12µ/s)
    11.3 Washing with water. Transfer the waste tube to the water waste bottle
    11.4 Do task - Nozzle removal wash
        - Disconnect the PDC and connect the flush bottle to the manifold washing with water. Robot setup-> Syringe pump.(Volume 4000µl, Speed 40µl/s)
    11.5 Washing and removing PDC:
        - Connect the PDC to the manifold when the first drop appears
        - In tab Do task choose Wash flash strong narrow
        - For check the droplet press Continuous Dispensing bottom
        - In tab Do task choose Nozzle removal wash
    11.6 As relevant (if the system is not in use on the next day) go to Do task and perform Dry system
12. Power off the system: SciFlex_s3 program, PC, dry air insertion, vacuum pump, remove Well384.

Supplementary Manual 3| Permeation Oven operation

The permeation oven CGM 2000 consists of three ovens. Each oven works separately, and its temperature and properties are controllable individually. The temperature ranges for the first and second ovens are: 30°C-110°C and the third oven ranges between: 10°C-110°C. Each oven should include one diffusion tube. There are three diameter sizes for the diffusion tubes (0.5, 2.2 and 5 mm).

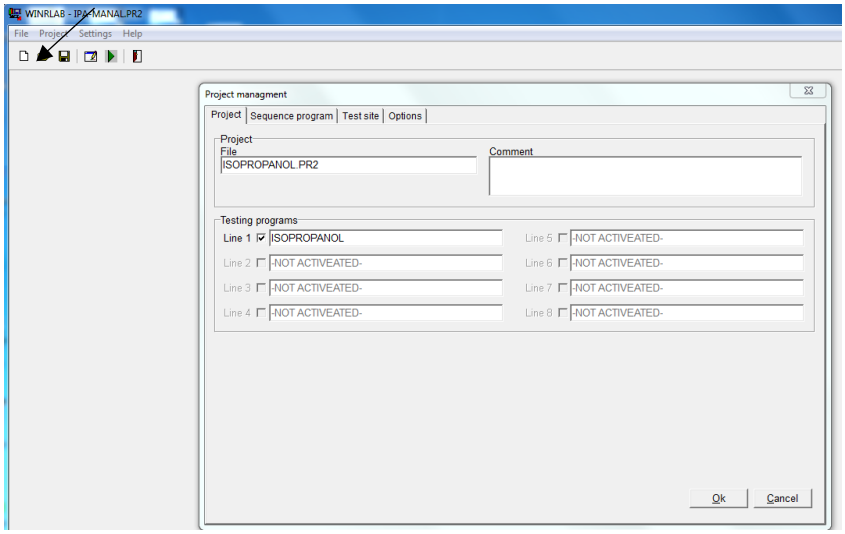
To reach the required concentration, calculate the following parameters:

Boilling Temp. @ 1 atm (ºC)	literature source.
Boilling Temp. Limit (ºC)	5ºC below boilling temp. of liquid (at 1 atm).
Oven Temp. (ºC)	Must be <=110C and <= Boilling_Temp._Limit
Diffusion Coeff. @ 25ºC (cm^2/sec)	Acc. "Diffusion Coefficients.PDF" or other literature source.
MW (gr/mol)	literature source.
Capillary ID 0.5 or 2.2 or 5 (mm)	User Definable (0.5,2.2,5)
Cross Sectional Area (cm^2)	PI()*(Capillary ID*0.1/2)^2
Length of Diffusion Path (cm)	Constanst = 6.8
Atmospheric Pressure (mmHg)	Constanst = 760
Vapor Pressure (mmHg)	calculate using Antoine equation , acc. to "Knovel- Yaws' Handbook of Thermodynamic and Physical Properties of Chemical Compounds: Vapor Pressure"
Diffusion Rate (ng/min)	19000*(273.15+Perm-Temp)*Diffusion Coefficient *Molecular weight*(Area [cm2]/B4)*LOG10(ambient pressure/(ambient pressure-vapor pressure))
Molar Const.	22.4/MW (gr/mol)
Min Conc. (ppm)	Diffusion Rate (ng/min)*Molar Const./10000
Max Conc. (ppm)	Diffusion Rate (ng/min)*Molar Const./1000

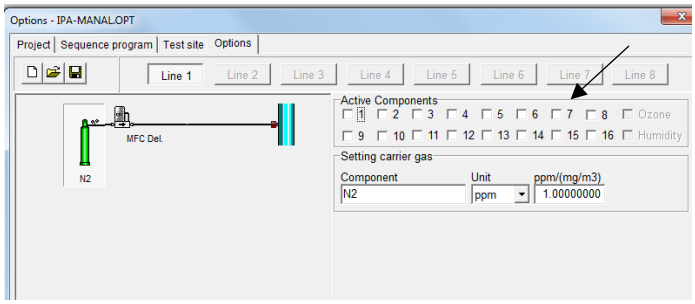
Operation of calibration system- Model CGM 2000 (Serial no.:1101-011)

1. Start the program. (CGM 2000)

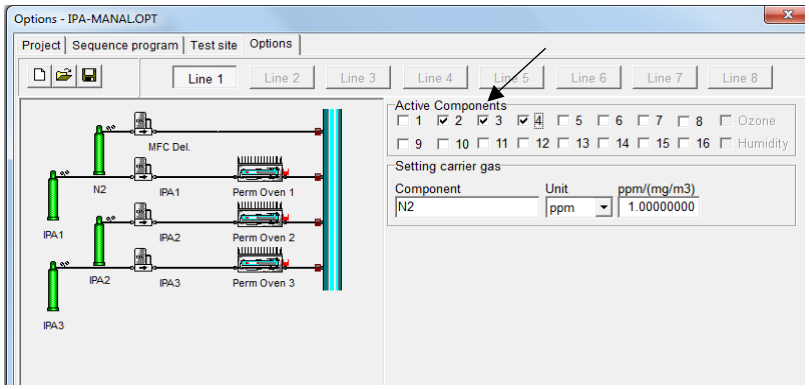
2. Start project management: Write the project file name. This name and the testing program name must be the same name.



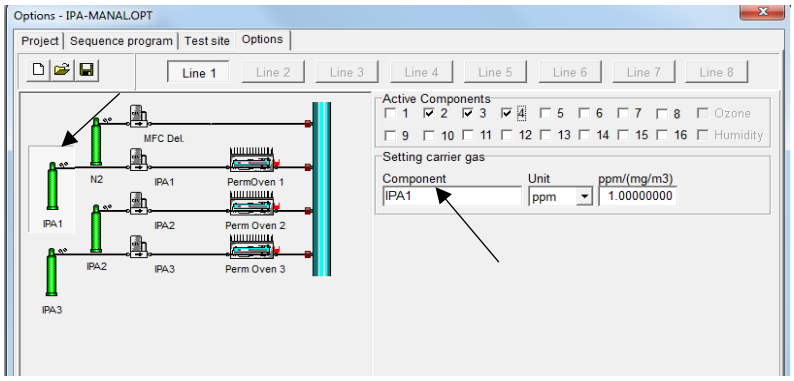
3. Options menu: Choose the active components you need (choose which ovens to use)



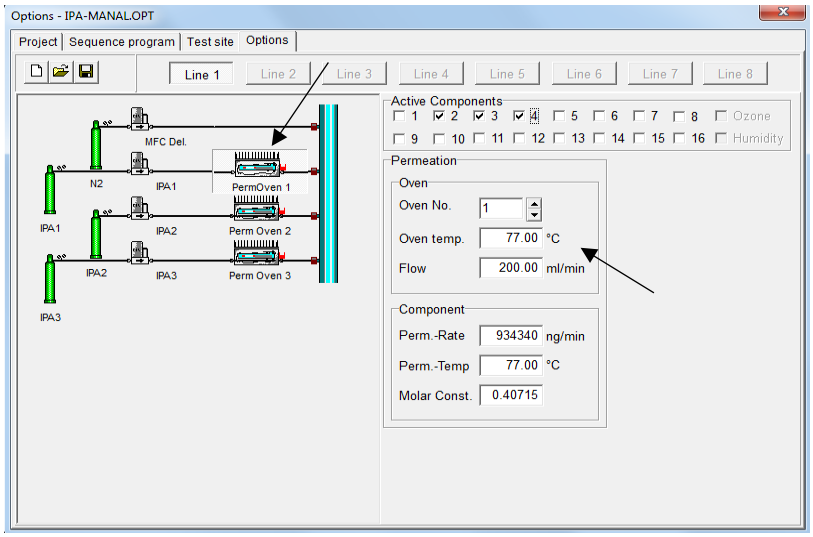
4. Choose 2, 3, and 4 to work with the three ovens.



5. Press on the green tube at the left of the window and write the component name.

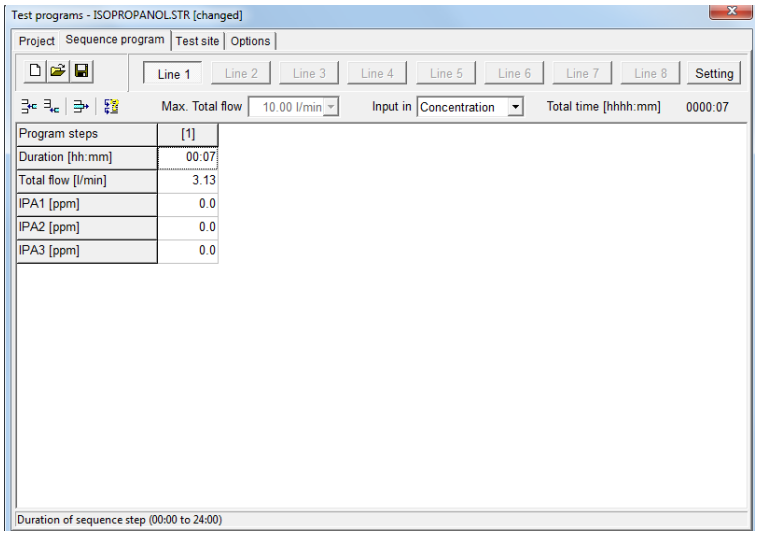


6. Press on the PermOven to write the parameters which you calculated before: Temperature, molar const. and Perm.-rate

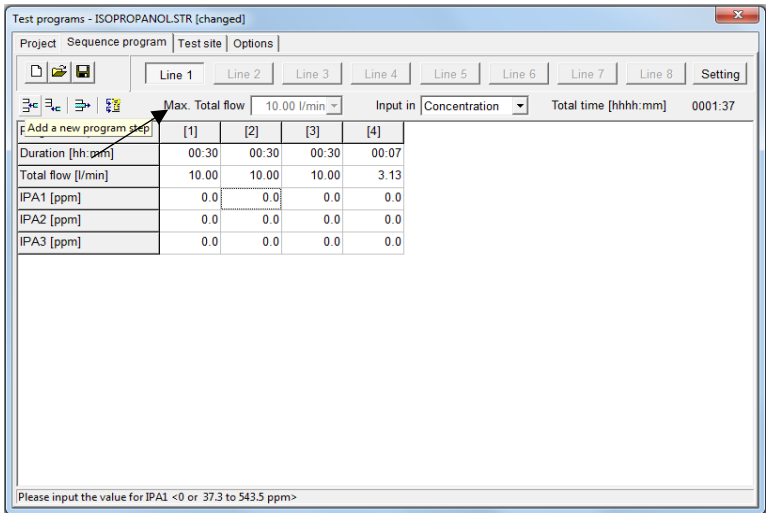


7. Save the program in this step. The name must be the same as you called it at the beginning.

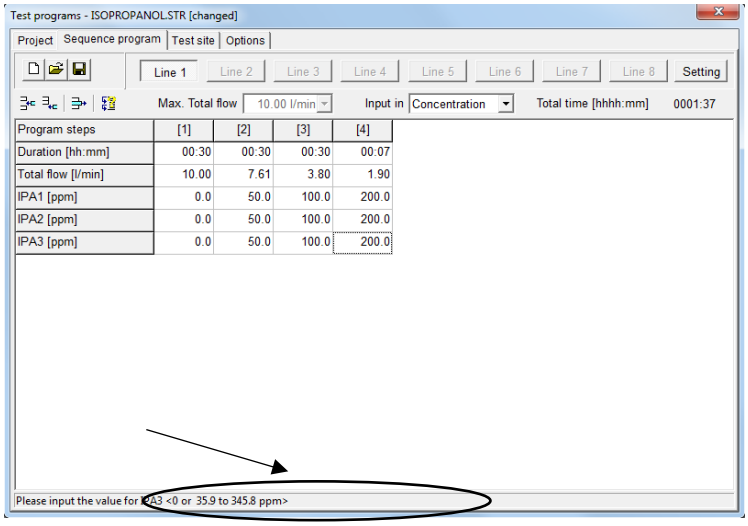
8. Go to sequence program.



9. Add a new program step.

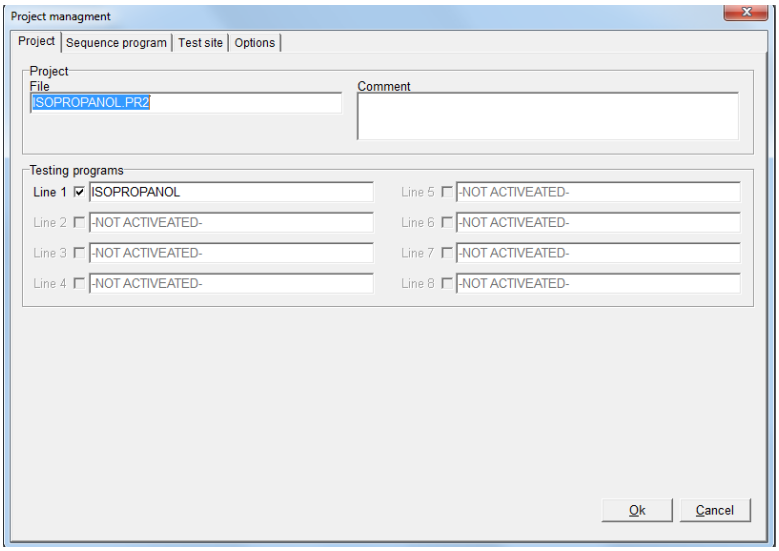


10. Choose a concentration for each oven in each step. The concentrations that you can set based on the parameters you have defined are written down. You can change duration of each step.

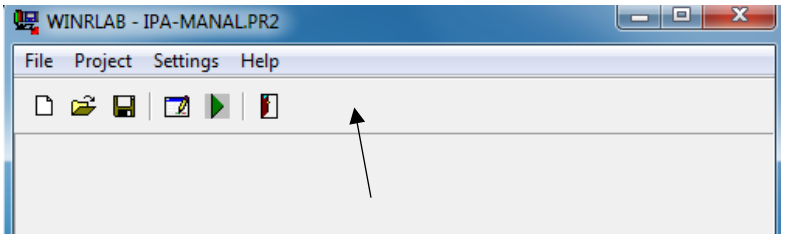


11. Save the program again with the same name you have saved before.

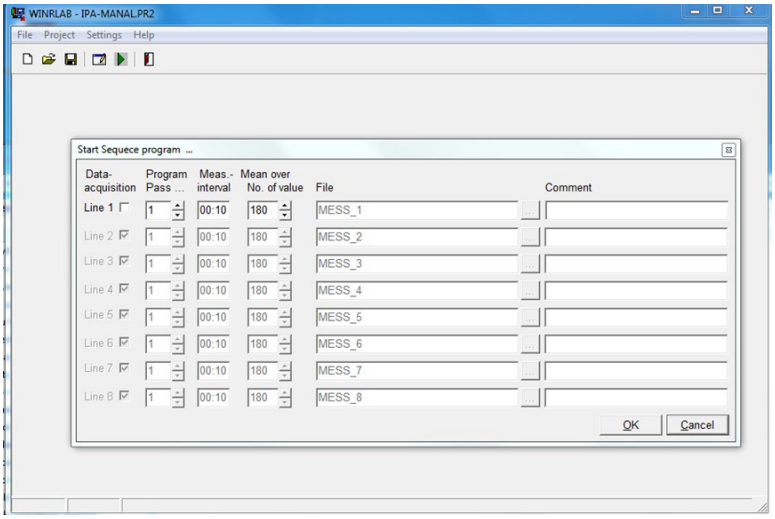
12. Go to project and press OK.



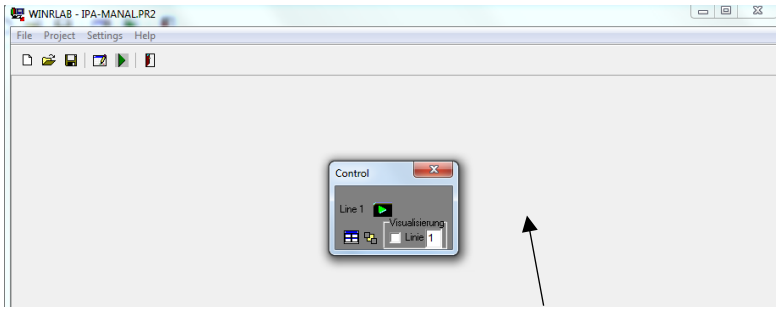
13. Click on the play



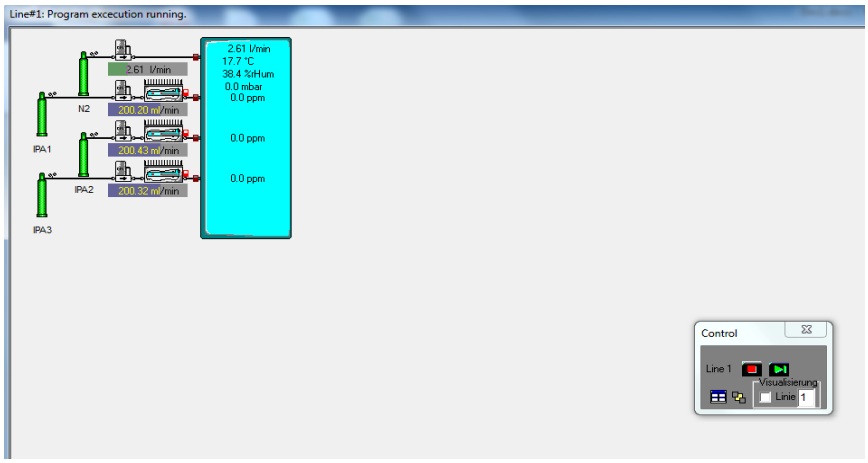
14. Click OK in the window that appears.



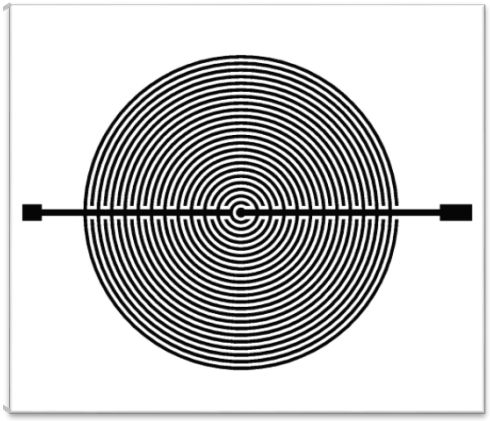
15. Start running the program –>play.



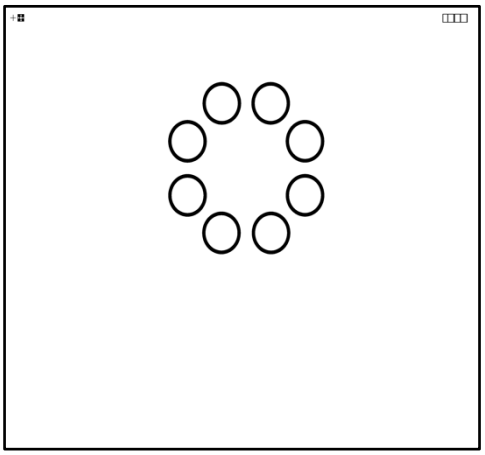
16. The gas will start to flow as you defined.



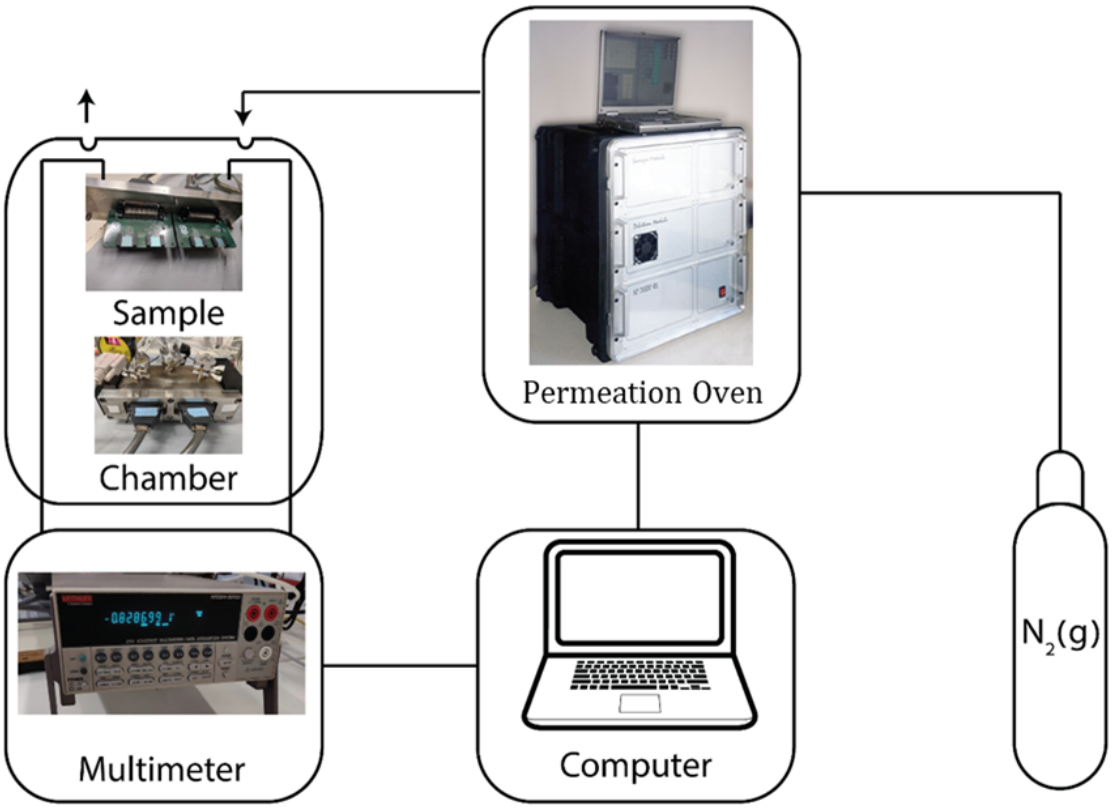
Supplementary Figures



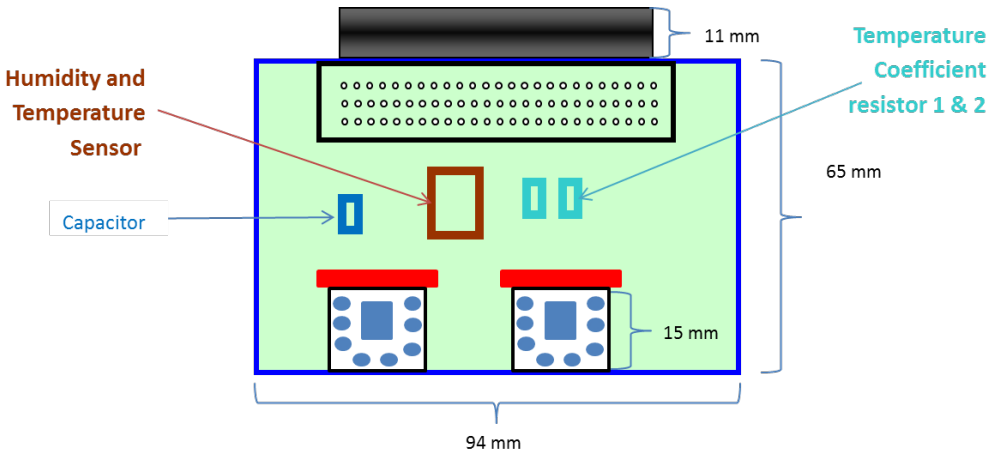
Supplementary Fig. 1: The electrode mask scheme is a first step



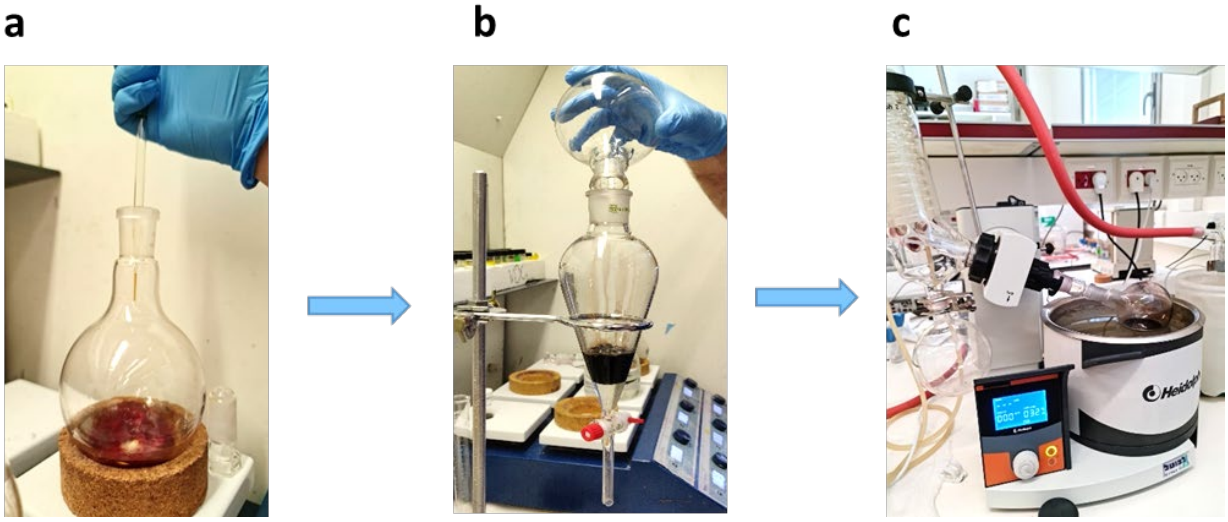
Supplementary Fig. 2: The micro-barrier mask is a second step



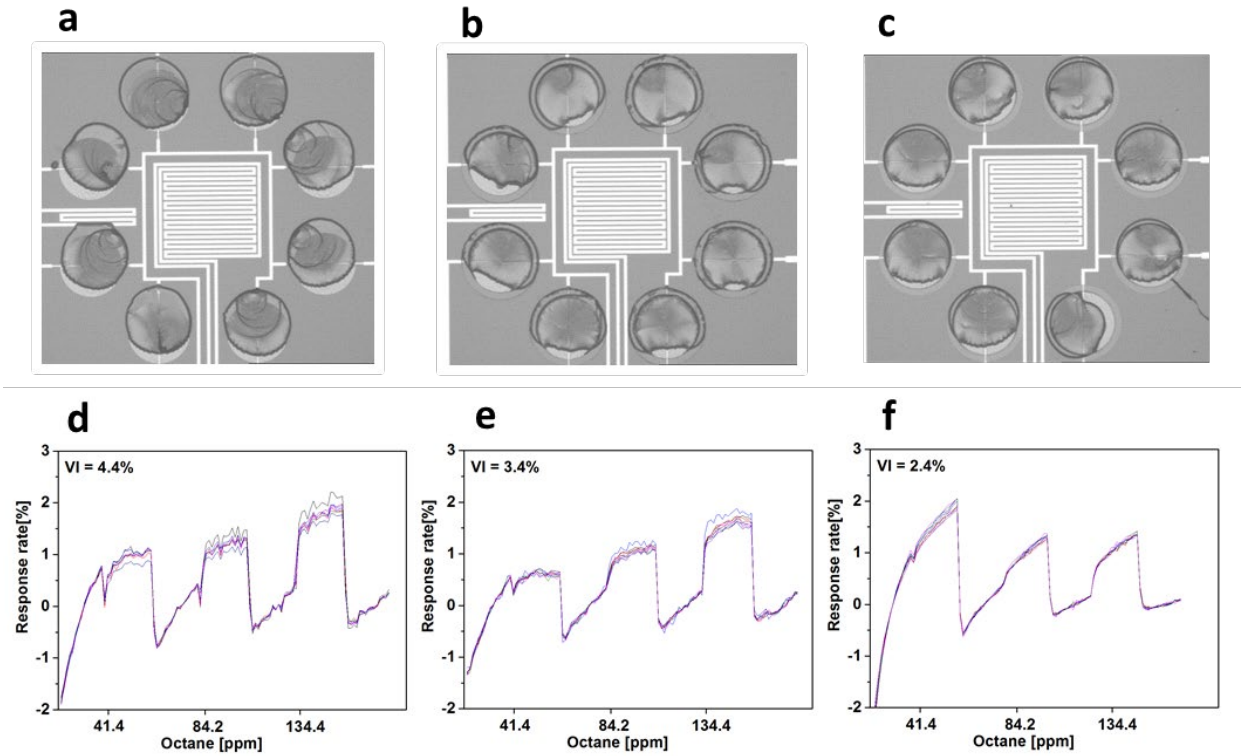
Supplementary Fig. 3: The setup for the sensors response examination. The devices are inserted into the PCB in a stainless-steel chamber with a volume of 100 cm^3 and the electrical resistance of the sensors is measured by a multimeter (Keithley 2701) when an analyte gas is injected into the chamber. The computer programs control the gas flow from permeation oven and the electrical measurements (see Supplementary Manual 2).



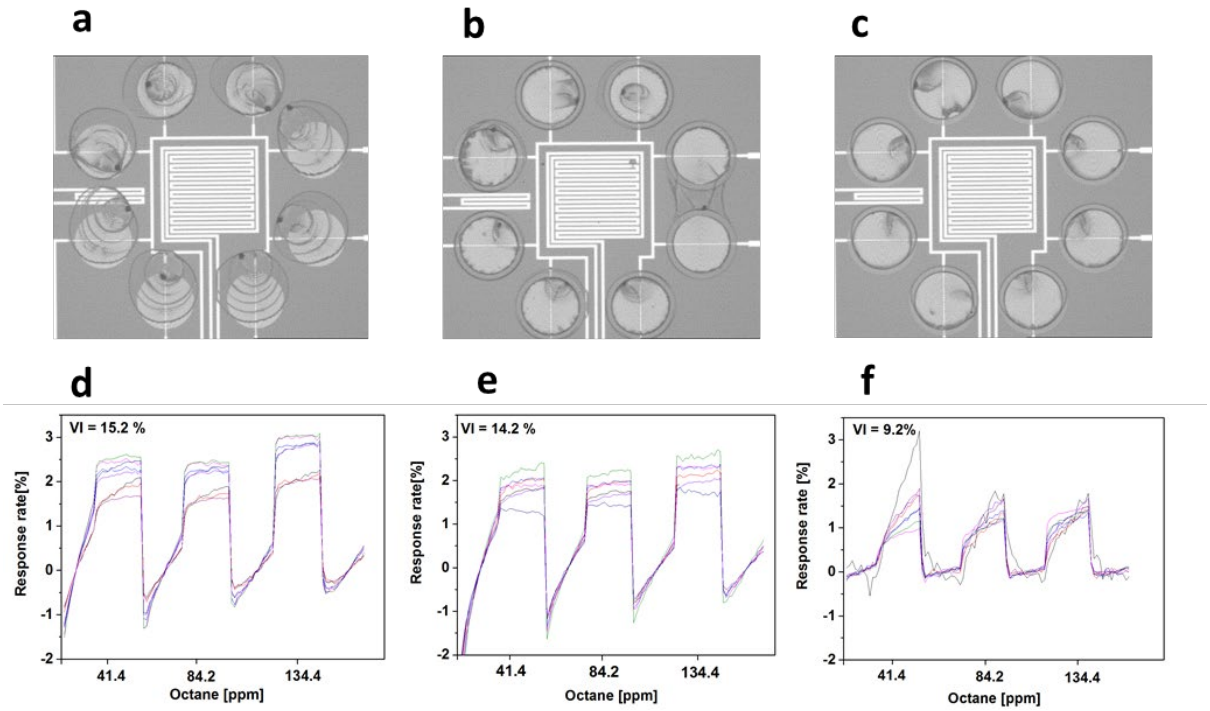
Supplementary Fig. 4: The schematic picture of the PCB. The connector of the PCB (CAS no. 09-66-552-7611) Sockets D-Sub Connector Right Angle 50 Way,HARTING Manufacturer



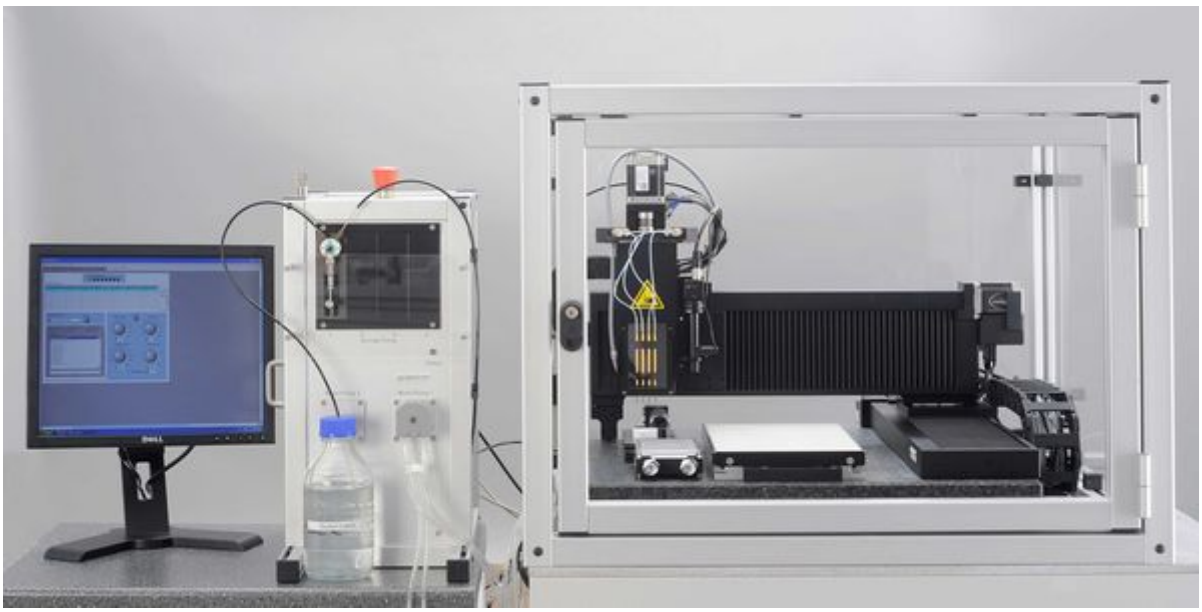
Supplementary Fig. 5: Pictures of synthesis and purification steps: **(a)** GNPs synthesis step in an around bottom flask while adding Thiols reagents, **(b)** phase separation step NPs in Toluene from water phase, **(c)** Toluene evaporation step using rotary evaporator.



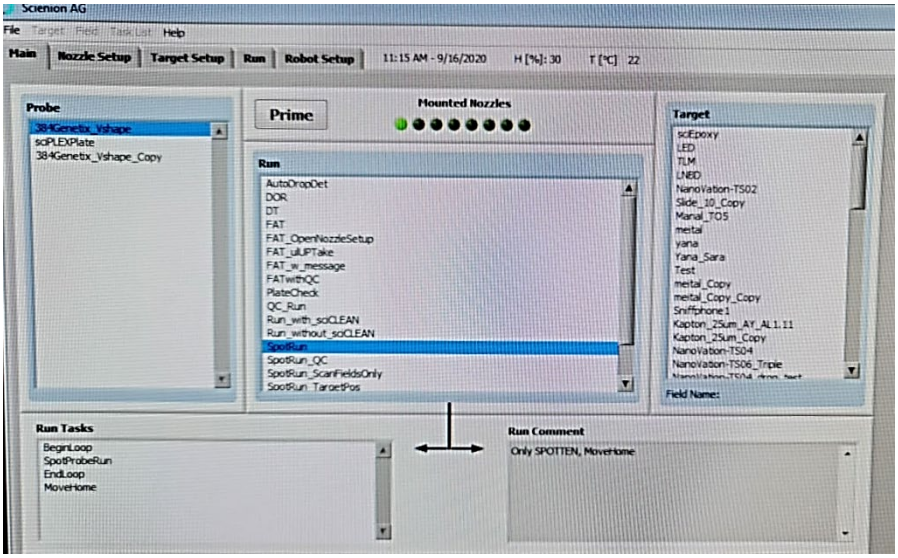
Supplementary Fig. 6: Comparison of sensors response when prepared by different techniques: **(a)** Without any barrier. **(b)** With SiO2 ring barrier. **(c)** With SU-8 ring barrier. The GNP printed volume is 20nl. The type of GNP is 2-Ethlhexanethiol 20mg/ml. The response to octane was measured after drying in vacuum oven for 25h. **(d)** The response of the chip without any barrier. **(e)** The response to the chip with SiO2 ring barrier. **(f)** The response on octane of the chip with SU8 ring barrier.



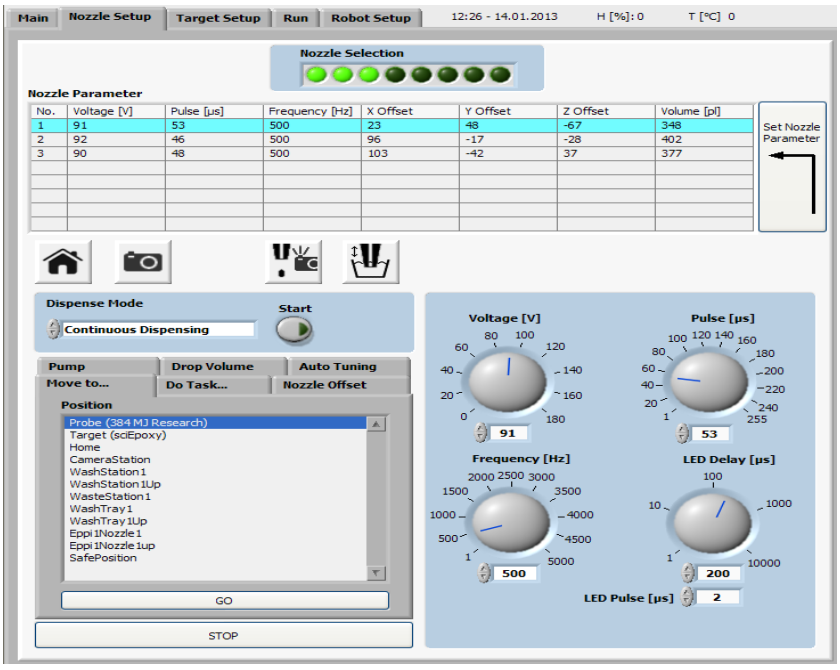
Supplementary Fig. 7: Comparison of sensors response when prepared by different techniques: **(a)** without any barrier. **(b)** With SiO2 ring barrier. **(c)** With SU8 ring barrier. The GNP drop casting volume is 20nl. The sensing layer is based on is dodecanethiol modification in concentration of 5 mg/ml. The response to octane was measured after drying in vacuum oven for 45h. **(d)** The response of the chip without any barrier. **(e)** The response to the chip with SiO2 ring barrier. **(f)** The response of the chip with SU8 ring barrier.



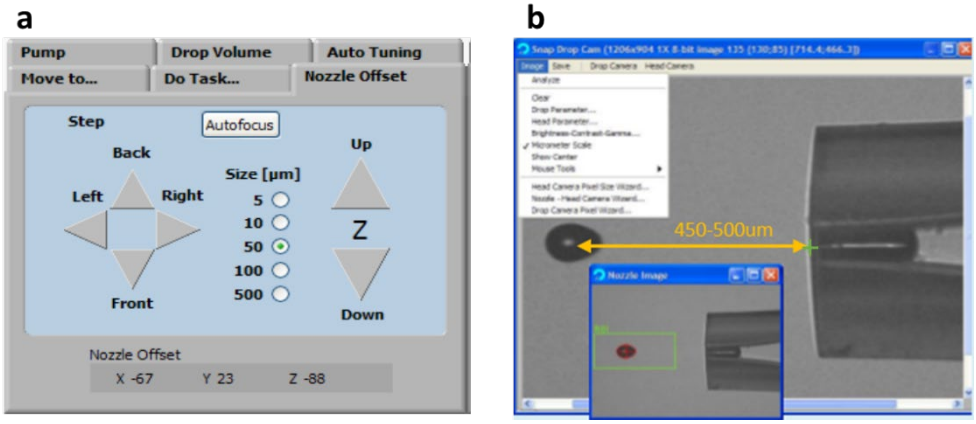
Supplementary Fig. 8 Automated piezo driven, non-contact dispensing system - Scienion sciFLEXARRAYER S3.



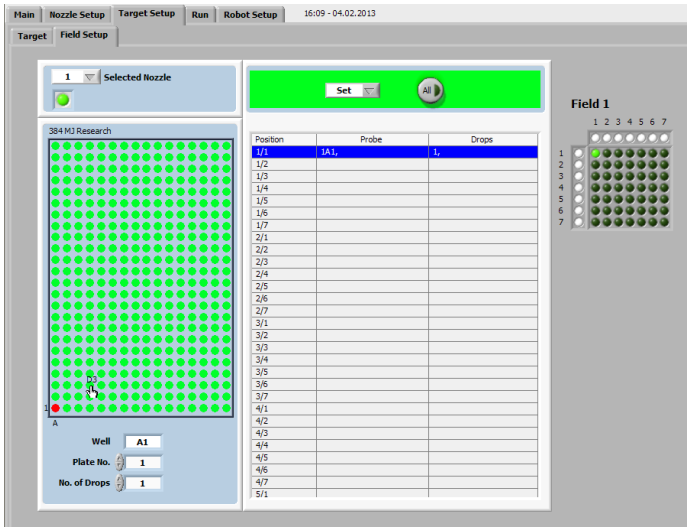
Supplementary Fig. 9: The main operation menu of the dispensing system. This main window allows the user to select the probe type (where the samples come from) and target type (where the samples have to go) and to choose pre-written runs.



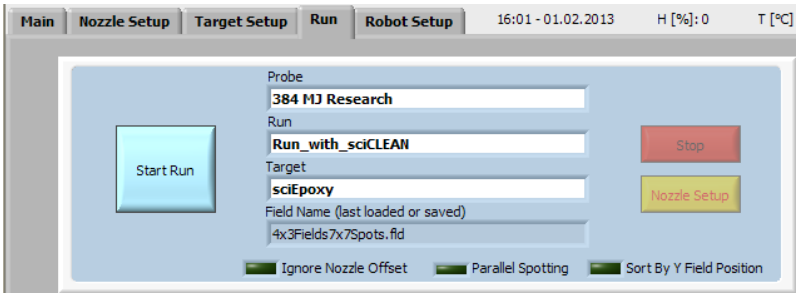
Supplementary Fig. 10: Nozzle parameters setup. It allows control for the nozzle parameters including the number and positions of the nozzles installed, the optimized nozzle parameter settings etc., and the option to move to the predefined positions and perform the pre-written tasks.



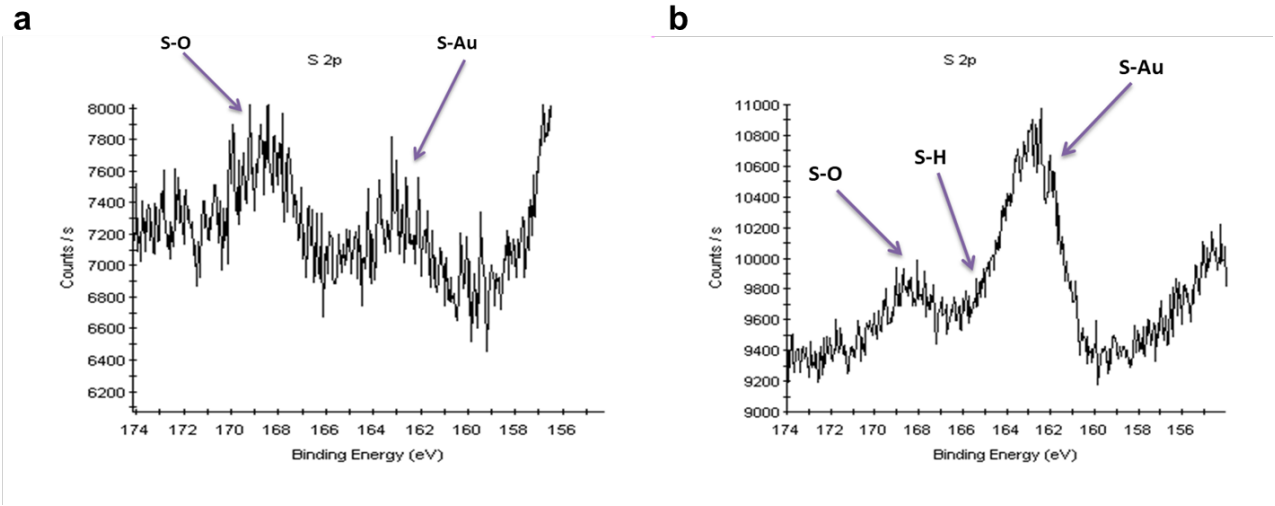
Supplementary Fig. 11: Setting of the nozzle windows; **a)** nozzle offset window; **b)** the edge of the nozzle and distance determination between drop and the center of nozzle.



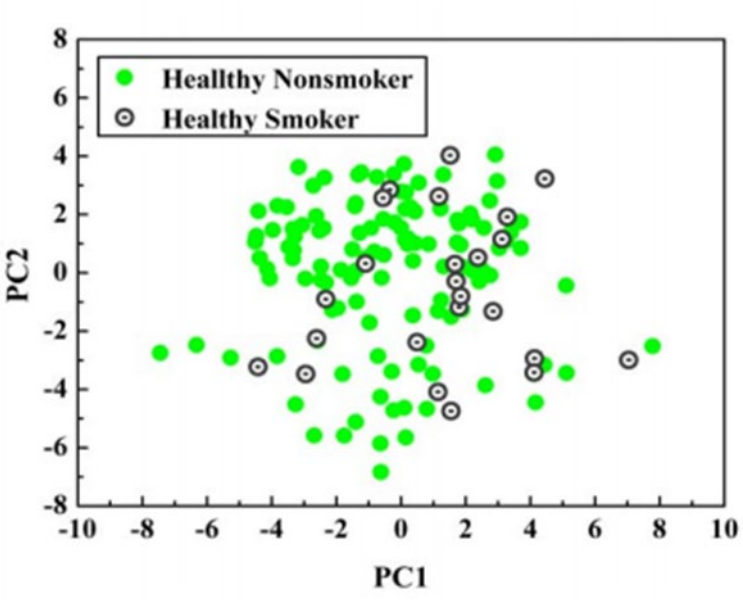
Supplementary Fig. 12: Field setup window**.**



Supplementary Fig. 13: Run setup window. Here a prewritten run can be started and stopped. The progress of the run and the samples processed are indicated during the run procedure.



Supplementary Figure 14: XPS characterization of dodecanethiol caped GNP on SiO2 surface where **(a)** and **(b)** are the high-resolution XPS spectrum of S2p of thin (0.3µm) and tick (2.5µm) sensing film respectively. The thin GNP film has a different surface composition in comparison the thick film.



Supplementary Figure 15: Principal component analysis (PCA) of the multidimensional ΔR/Rb data set of real breath of 16 smoking volunteers and 40 nonsmoking healthy volunteers. Reprinted from ref. 1 with permission from Nature Publishing Group, copyright 2009.

Reference:

1. Peng, G.*, et al.* Diagnosing lung cancer in exhaled breath using gold nanoparticles. *Nat. Nanotechnol.* **4**, 669-673 (2009).