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pH SENSING WITH A GFET-PV01

Application Note Series #1 from the Paragraf Technical Centres

The measurement of the partial pressure of hydrogen ions (pH) is not novel, but its measurement forms the basis of many product formulations and Quality Control parameters for product release. pH measurements under more challenging conditions remains a need of certain industries including food and beverage, etc., as well as applications where space or power constraints remain a consideration.

Graphene Field Effect Transistors (GFETs) offer the potential solution to these unmet needs, with excellent dynamic range, resolution and sensitivity, and small geometry. This application note provides feasibility evidence of performance in two structural formats, first as bare graphene sensors where the channels remain directly exposed to the solution, then as sensors capped with a metal oxide that provides additional sensitivity and selectivity for hydrogen ions.

While the experiments herein are provided for application interest, they do not, and are not intended to represent optimised conditions nor optimised device designs. The GFET-PV01 is a research-focussed transistor, with just three electrode channels equally spaced around a large central gate that are distanced from one another to permit manual adjustment or adjustment with automated targeted deposition. Pristine graphene devices (flat, genuinely monolayer, free from polymer or metal contamination) were used straight from stock and represent performance that can be achieved by any scientist without additional equipment.

Paragraf GFETs are the only commercially scalable graphene devices that are manufactured on semi-conductor style processes and equipment, offering unparalleled quality and consistency. The metal oxide (Hafnium) capped devices are an internal product variant not currently on sale. These research prototypes were produced by Paragraf's Foundry using patented technology and knowhow in 2D materials. Experiments conducted were performed with appropriate rigour and attention to detail, within a controlled experimental environment, and used buffer standards that stretch device parameters. They were not intended to mimic any particular product or application environment other than laboratory use.

The data shared herein, is intended to serve as evidence of use case, and exemplification that can be replicated independently and further built upon to establish specific product or application alternatives that meet specific needs or research interests.

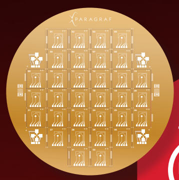
Please express your interest in these devices through sales@paragraf.com. Limited numbers may be accessible as engineering prototypes on terms where performance data is shared.

If sufficient demand exists for the metal oxide modified GFET devices, Paragraf will expedite the routine production and include them in the catalogue, otherwise these devices will remain available by bespoke order with a modest minimum volume requirement. Unmodified GFET-PV01 devices are on sale and in stock now.

Further application notes, covering gas sensing and other exciting applications for GFETs are available from Paragraf. Email sales@paragraf.com for further information.

EXPERIMENTAL METHODS AND RESULTS

GFET-PV01 devices were used for all experiments, at room temperature.



100mM stock buffer solutions were prepared from 1M Potassium Phosphate Monobasic and 1M Potassium Phosphate Dibasic solutions (Sigma-Aldrich) at the different ratios. Very basic solutions were adjusted with 1M NaOH solution (Alfa Aesar). Very acidic solutions were adjusted with 1M Phosphoric Acid (Honewell). pH validated with calibrated pH electrode (ThermoScientific, Orion Star A211)

All stock buffers were further adjusted to final working solutions containing 9mM Potassium Chloride and 10mM Phosphate buffer. pH validated, again.

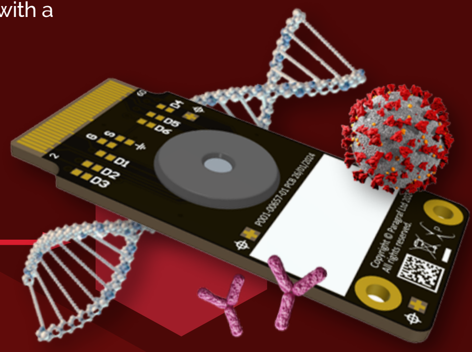
The gate electrode was modified with Ag/AgCl ink (EIC, 60/40) via a 'hanging drop' at the end of a ½" needle and direct contact with the gate to cover the entire electrode surface and oven to dried for 10min at 130°C.

All solutions were added to GFET-PV01 using a Gilson pipette P200.

All pH measurement cascades were started at pH7.15, whereby the first buffer addition from dry state (packaging) was given 120 seconds equilibration before measurement. Thereafter, each buffer exchange was measured for just 60 seconds before replacing with a subsequent buffer.

30µl of each buffer solution was applied to the sensor well of each GFET-PV01, and removed by automatic handheld aspirator (Integra, Vacusip). No washing step between sequential samples.

GFET-PV01 devices were connected to 2x Keithley 2450 Source Measure Units (SMU) with custom-built multiplexer device. The Dirac Point was determined using the Paragraf bespoke "Dirac Point Tracking" software (to be released soon).



For the Hafnium Oxide capped prototype devices, Hafnium Oxide was deposited directly on top of finished GFET-PV01 devices at wafer level, prior to scribing, dicing and packaging. An e-beam deposition process was used to deposit a hafnium oxide layer on top of a layer of aluminium oxide across the graphene wafer surface in a non-optimised manner. Die (capped) were then incorporated into the manufacturing process resulting in identical GFET-PV01 device architecture. The Ag/AgCl gate was applied in the same manner as for standard GFET-PV01 devices.

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GFET-PV01 SPECIFICATION

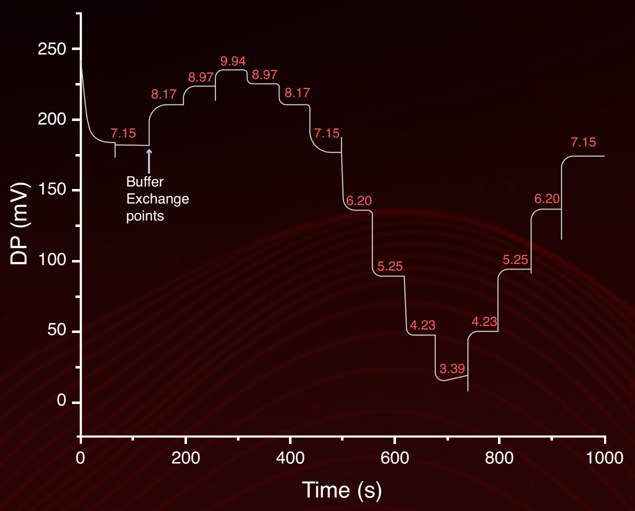

Figure 1. An exemplar pH titration cascade (one of three performed, using different 3 different GFET-PV01 devices) using 10mM Phosphate Buffer with 9mM KCl at room temperature. GFET-PV01 devices were given 120 seconds to equilibrate from dry state, whereas all subsequent measurements were given just 60 seconds between exchanges. The graph shows the non-linear but consistent change in Dirac Point with shift in pH.

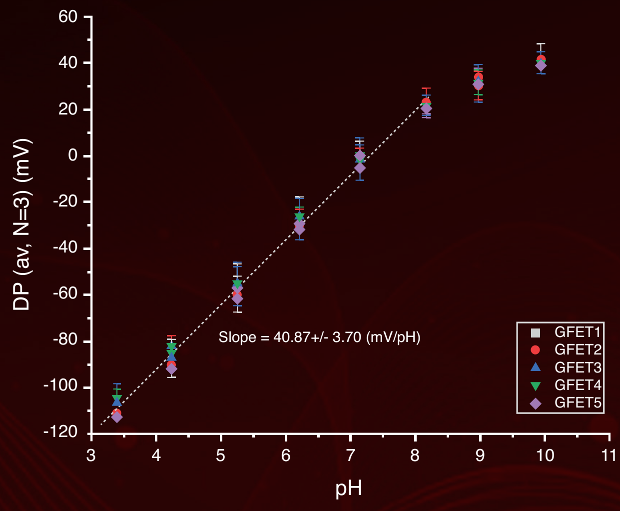

Figure 2. Schematic representation of typical data resulting from the measurement of pH using pristine GFET-PV01 sensors from buffer standards at the values shown across the X-Axis. Each data point is representative of an average of 3 independent titrations series. Measurements were made at the Dirac Point (with respect to Ag/AgCl gate), normalised to pH 7.15 which is used as a control throughout the tests. Data from 5 GFET channels is presented.

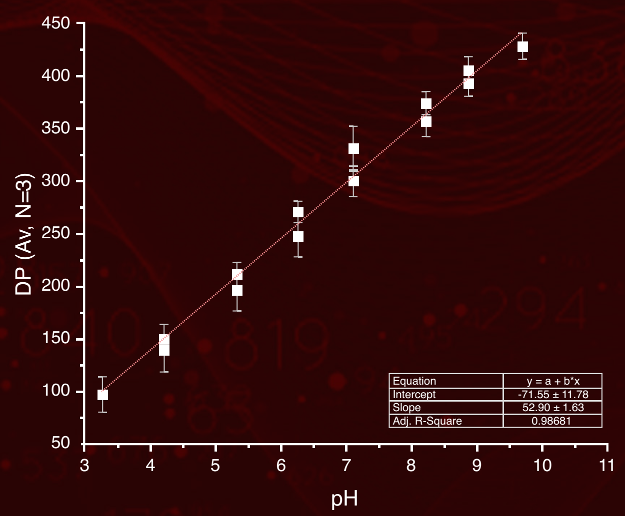

Figure 3. Schematic representation of typical data resulting from the measurement of pH using Hafnium Oxide capped GFET-PV01 sensors from buffer standards at the values shown across the X-Axis. Each data point is representative of an average of 3 measurements at each point, made concurrently before replacing with the subsequent buffer. Measurements were made at the Dirac Point, as shown on the Y-Axis.

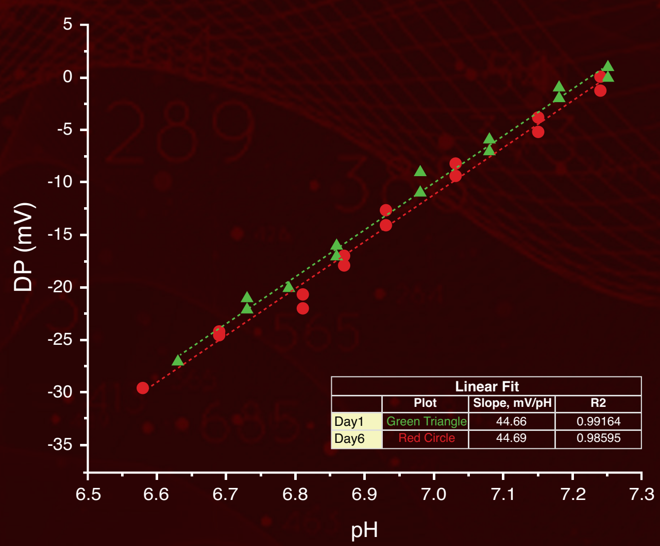

Figure 4. The above example shows the continued pH correlation with shift in Dirac Point over multiple sequential days, confirming reproducibility and reusability. The data also shows the finer resolution which is achievable with the current GFET devices in a non-optimised format.

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