

Knowledge Representation of Ion-Sensitive Field-Effect Transistor Voltage Response for Potassium Ion Concentration Detection in Mixed Potassium/Ammonium Ion Solutions

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Abstract: Problem statement: The Ion-Sensitive Field-Effect Transistor (ISFET) is a metal-oxide field-effect transistor-based sensor that reacts to ionic activity at the electrolyte/membrane/gate interface. The ionic sensor faces issue of selectivity from interfering ions that contribute to the sensor electrical response in mixed solutions. **Approach:** We present the training data collection of ISFET voltage response for the purpose of post-processing stage neural network supervised learning. The role of the neural network is to estimate the main ionic activity from the interfering ion contribution in mixed solutions given time-independent input voltages. In this work, potassium ion (K^+) and ammonium ion (NH_4^+) ISFET response data are collected with readout interface circuit that maintains constant voltage and current bias levels to the ISFET drain-source terminals. Sample solutions are prepared by keeping the main ion concentration fixed while the activity of an interfering ion varied based on the fixed interference method. **Results:** Sensor demonstrates linear relationship to the ion concentration within detection limit but has low repeatability of 0.52 regression factor and 0.16 mean squared error between similarly repeated measurements. We find that referencing the voltage response to the sensor response in DIW prior to measurement significantly improves the repeatability by 15.5% for correlation and 98.3% for MSE. Demonstration of multilayer perceptron feed-forward neural network estimation of ionic concentration from the data collection shows a recognition of >0.8 regression factor. **Conclusion:** Time-independent DC voltage response of ISFET of the proposed setup can be used as training data for neural network supervised learning for the estimation of K^+ in mixed K^+/NH_4^+ solutions.

Key words: Microsensors, electrochemical devices, back-propagation, supervised learning, selectivity

INTRODUCTION

The ion-sensitive field-effect transistor (ISFET) is an electrochemical sensor that produces electrical response in accordance to ionic activity at the exposed gate window (Bergveld, 1991; 2003a). Similar to the more frequently used ion-selective electrode (ISE), ISFETs are potentiometric sensors that produce response by virtue of potential reaction at the electrolyte/membrane interface (Izquierdo and María, 1995; Cobben *et al.*, 1994). Unlike its ISE counterpart, the structure of ISFET is similar to the metal-oxide field-effect (MOSFET) transistor thus giving ISFET the advantage of being solid-state silicon based and compatible with standard MOSFET fabrication technology (Bergveld, 2003a; Bergveld, 1985;

Chin, *et al.*, 2001). Ion-sensitive sensors find wide applications in liquid analysis (Humenyuk *et al.*, 2006; Gieling *et al.*, 2000; Moreno *et al.*, 2006), microbiology (Kharitonov *et al.*, 2000; Swaminathan *et al.*, 2002; Castellarnau *et al.*, 2008) and agriculture (Gieling and van den Vlekkert, 1996; Gieling *et al.*, 2005) applications.

In the presence of mixed ions of similar charge type and number along with the main ion of interest, ISFETs exhibit response towards the interfering ionic activity. The sensor signal in mixed-ion solution represents combined activity rather than information from a single ion (Leistiko, 1978; Deyhimi, 1999).

The common method in finding the selectivity coefficient is provided by the International Union of Pure and Applied Chemistry (IUPAC) standard for ISE

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that is applicable to ion-sensitive field-effect transistors (Bratov *et al.*, 2004). The approach involves parameter estimation and interpolation of chemical data.

Post-processing stage involving machine learning has been proposed to estimate ionic concentration change and to extract the main ion activity from the mixed response such as blind source separation technique (Bedoya *et al.*, 2004; Bermejo and Sole-Casals, 2004; Bermejo *et al.*, 2003; Bermejo *et al.*, 2006) that requires time-dependent voltage response. Pattern recognition methods are also used with an array of sensors providing a series of input features for classification (de Sousa *et al.*, 2002).

In this study, ISFET voltage response is obtained for the purpose of recognizing the potassium ion (K^+) logarithmic value of concentration in the presence of ammonium ion (NH_4^+) in ranges of concentration level typical to agricultural surroundings. The measured data will function as training data intended for a post-processing stage neural network based on supervised learning. The sensor is biased with a constant drain-source current and voltage acting as the readout interface circuit. The scope of work covers from the point of chemical and electrical device setup up to post-processing feed-forward neural network.

MATERIALS AND METHODS

Training data is collected from K^+ and NH_4^+ sample solutions between -6 to -1 log of ion concentration by mixing potassium and ammonium chloride with De-Ionized Water (DIW). The chosen ions are known to be most interfering with each other having the same charge type and charge number. The goal of sample preparation is to represent the input space within the detection limit of available K^+ and NH_4^+ ISFET. The logarithmic values of potassium ion concentration (pK) and of ammonium ion concentration (pNH₄) are used; the intended target data format is {pK, pNH₄}. The electrical response for the particular target data is taken from an array of sensors through interfacing circuits that would function as time-independent input data {V₁, V₂, ..., V_m} to a neural network post-processing stage. The goal of the readout circuit is to monitor threshold voltage modification due to electrochemical effects by the electrolytes in the solution at the gate/membrane/electrolyte interface.

Sample preparation: Sample preparation is based on IUPAC recommendations for fixed interference method that varies one type of ion activity by adding titrants to a total solution while keeping the other ion unchanged

(Umezawa *et al.*, 1995; 2000). Required volumes in the titration process is calculated from dilution equation and the log of activity of cation, γ , based on the Debye-Huckel approximation (Skoog *et al.*, 2003):

$$I_{total} = \frac{V_o \cdot I_o + \sum_i V_{titrant_1,i} \cdot I_{titrant_1} + \sum_i V_{titrant_2,i} \cdot I_{titrant_2}}{V_{total}} \quad (1)$$

$$\log(\gamma) = - \left(\frac{0.509 * \sqrt{I_{total}}}{1 + (2.5 * 0.328 * \sqrt{I_{total}})} \right) \quad (2)$$

Where:

I = Ionic strength

V = Volume of solution

Subscripts:

o = For initial sample

titrant = Indicating added titrant

total = Indicating total solution

For K^+ , seven different levels of ionic strength initial solutions are prepared with {pK|pK $\in\mathbb{Z}$, -6 \leq pK \leq -1} and one control solution without K^+ introduced. Likewise, for NH_4^+ , seven initial solutions are prepared with {pNH₄|pNH₄ $\in\mathbb{Z}$, -6 \leq pNH₄ \leq -1} and one control solution without NH_4^+ . The second type of ion is added to each of the initial solutions 11 times by pre-calculated volume of titrants based on (1) and (2) successively of which some are control values outside sensor detection limits. This results in 12 possible values of varying pK in fixed pNH₄ and vice versa. This approach results in a total of 168 samples including 48 control samples.

Data acquisition: The drain-source ISFET would have to be biased with constant current and under constant voltage to allow the effects of the electrochemically induced voltage to be measured.

The ISFET is modeled with the electrical characteristics based on standard MOSFET drain current equation and the electrochemical effects included as threshold voltage modifications to the MOSFET device (Tomaszewski *et al.*, 2007; Bergveld, 2003b; Pierret, 1996) as in the following expression:

$$I_{ds} = \beta \left((V_{ref} - (V_{th} - E_{chem})) V_{ds} - \frac{V_{ds}^2}{2} \right) \quad (3)$$

Where:

I_{ds} = Drain current

β = Device parameter

V_{ref} = Applied reference electrode voltage

V_{th} = Threshold voltage without membrane
 E_{chem} = Electrochemically induced voltage
 V_{ds} = Drain-source voltage

Based on the concepts of electrochemistry, E_{chem} is related to ionic species as governed by Nernst and to the interfering ion by Nikolsky equation which is summarized into the following equation (Buck and Lindner, 1994):

$$E_{chem} = E_o \pm 2.303 \frac{RT}{z_i F} \log_{10} \left(a_i + \sum_j K_{ij} a_j^{z_i/z_j} \right) \quad (4)$$

Where:

- E_o = Potential at 1 mol dm⁻³ ionic activity
 - R = Gas constant
 - F = Faraday constant
 - T = Temperature
 - a = Ion activity
 - K_{ij} = Potentiometric selectivity coefficient
 - z = Charge numbers
- Subscripts:
- i = Main ion of interest
 - j = Interfering ion

The current-voltage transistor response of the sensor is initially investigated using semiconductor device characterization technique as shown in Fig. 1 to determine suitable operating point for the sensor in the readout circuit. The source and bulk are grounded, the reference electrode voltage bias is swept and the drain current is measured by the Source-Measure Units (SMU).

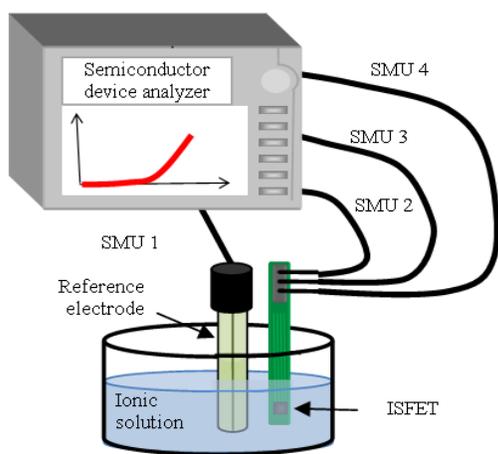


Fig. 1: ISFET and sample solution in device characterization setup

In order to monitor E_{chem} in (3), drain-source biasing has to be constant to focus on the effects of ionic activity change and the resulting electrochemically induced voltage. Based on isothermal point of operation, I_{ds} , V_{ref} and V_{ds} are set at 100 μ A, 0 V and 0.5 V respectively. The circuit implemented is shown in Fig. 2, with a variable resistor included to adjust the drain-source voltage bias. I_{ds} is held constant at 100 μ A by the current sources and the corresponding voltage is adjusted to be at 0.5 V_{ds} by varying the variable resistor attached to the output stage and fed back to the top amplifier (Abdullah *et al.*, 2009).

The circuit is implemented as shown in Fig. 3 allowing parallel measurements to be taken from an array of sensors. Since the reference electrode is connected to ground, a single electrode can be used for the array.

For continuous reading throughout the titration of one set on solution, the output of the interface circuit is fed to a data logger that communicates with the PC to record readings at 10 Hz sample rate as demonstrated in Fig. 4.

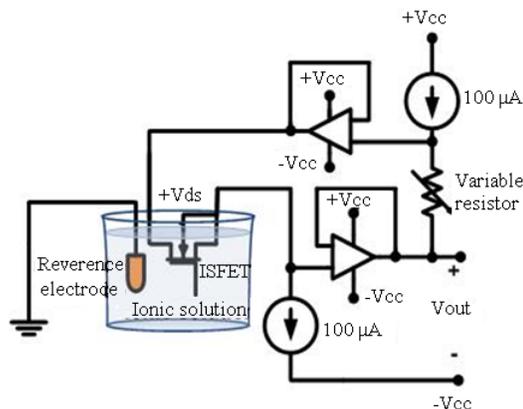


Fig. 2: Schematics of interface circuit for constant current-voltage drain-source operation (Gotoh *et al.*, 1989)

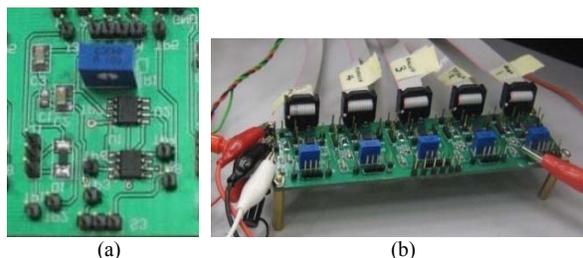


Fig. 3: (a) Implementation of readout interface circuit on PCB (b) Array of interface circuit for parallel measurements

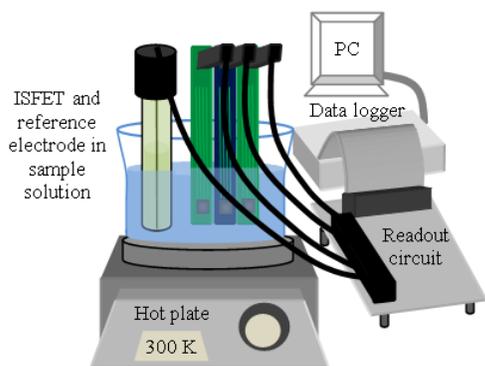


Fig. 4: Data acquisition setup

Feed-forward neural network: A multilayer perceptron architecture with a single hidden layer is built as post-processing stage for the sensor signals. The network is trained with supervised learning back-propagation algorithm. Training data size consist of 2-input dimension from sensor array of potassium and ammonium sensor with 2-output values of pK and pNH_4 . The hidden layer uses a hyperbolic tangent activation function. The activation function of the output neuron is linear. The synaptic weights linking the inputs to each neuron are adjusted according to the generalized delta rule with momentum. Training of the neural network employs stopping criteria based on absolute rate of change in the average squared error per epoch to be sufficiently small at 0.1%.

RESULTS

Semiconductor device behavior: From the semiconductor characterization setup in Fig. 2, ISFET device transfer characteristics are obtained with a sample shown in Fig. 5. In the higher ionic activity solution, -2 pK, the device has lower threshold voltage by 0.2 V, compared to the lower ionic activity -5 pK. The threshold voltage shifts the gate voltage corresponding to 100 μA drain current that will be applied in the circuit implementation by approximately 45 mV per order of concentration.

Data acquisition: Figure 6 demonstrates the measured sensor response starting from an initial solution and 11 subsequent additions of titrants for every 100 sec. One set of measurement gives 12 target samples taken over 1200 sec. The two plots shown differ in the dissolved ion content in the initial solution; one ionic strength of -4 p NH_4 and the other is the control set with no NH_4^+ introduced. For each of the 12 levels, an average reading is taken to represent a sample in the form of static value to represent {pK, p NH_4 }.

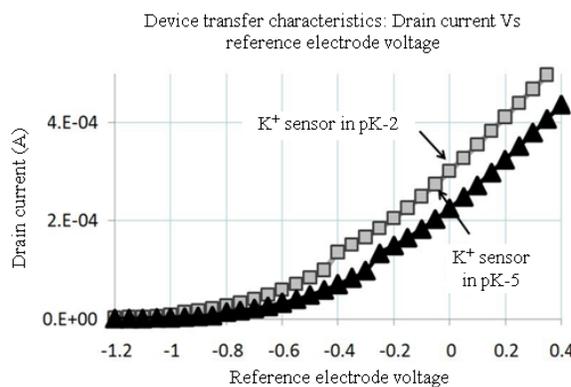


Fig. 5: Transfer characteristics demonstrating shift in threshold voltage in different levels of ionic activity

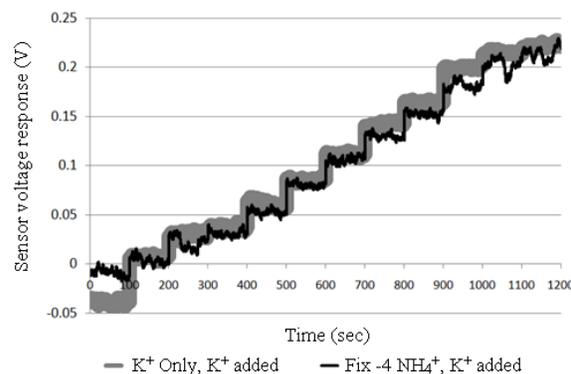


Fig. 6: Voltage response Vs time during titration of one set of fixed ion concentration level

Figure 7 shows the sensitivity of a K^+ sensor towards pK change in two differing interfering ion concentration in the background, one without NH_4^+ ion and another with -2 NH_4^+ ion in the background. With the presence of the higher interfering ion activity, lower detection limit of the main ion is increased thus limiting detection range. Without NH_4^+ in the background, the sensor detects as low as -5.5 pK. However, as expected, with -2 p NH_4 , linearity starts from -3.5 pK making the sensor less responsive to the ion it should be detecting. Figure 8 demonstrates the K^+ sensor response towards p NH_4 change in 6 different sets of fixed pK. Figure 8 indicates that the K^+ sensor is responding to the interfering ion especially when the interfering ion approaches the order of the main ion concentration. This means that if the main ionic activity is not strong enough, the sensor would be detecting other presence of interfering ion.

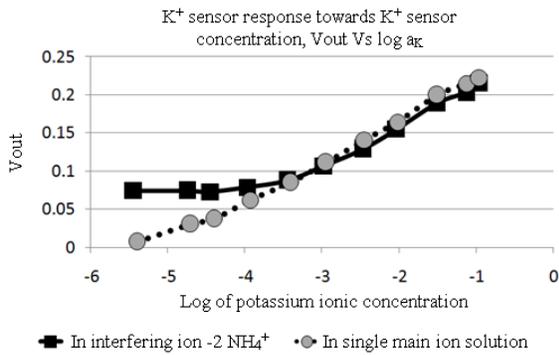


Fig. 7: Potassium sensor voltage response towards potassium ion concentration change in two different solutions

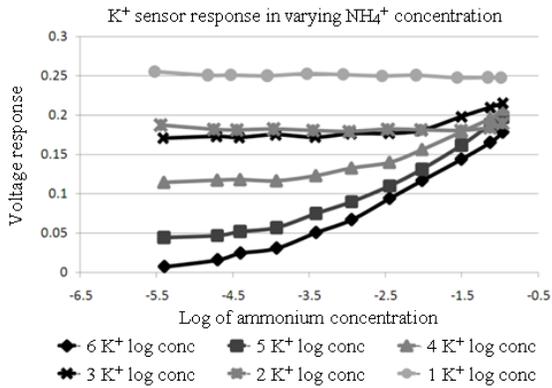


Fig. 8: K^+ sensor behavior in fixed potassium solutions between -6 to -1 log of K^+ concentration with varying ammonium concentration as interfering ion

DISCUSSION

Repeatability issue: Repeated measurements show that the sensor response demonstrates slight variation due to many possible sources such as light, temperature, device fabrication, membrane characteristics, membrane lifetime, reference electrode behavior and ambient electrical noise. It is also found that despite keeping the controllable factors such as light and temperature constant, the sensor demonstrates sudden DC voltage shifts as large as 0.7 V with subsequent readings maintained at this level.

The DC shifts is also observed in data extracted from device transfer characteristics setup in Fig. 2. A shift of 0.15 V in threshold voltage not accounted by ionic activity change is observed from device transfer characteristics despite having conditioned the sensor sufficiently. Figure 9 demonstrates the shifts in gate voltages for samples arranged in sets of gradually

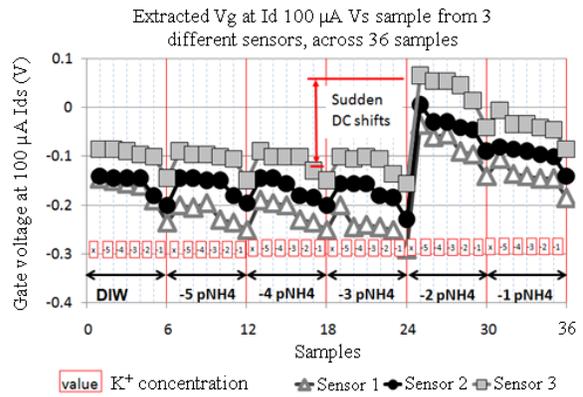


Fig. 9: DC shifts seen in the extracted data from the device transfer characteristics

increasing pK in groups of fixed pNH₄. Each data point corresponds to gate voltages required to reach 100 μA drain current, extracted by low-pass interpolation of the measured I_{ds} Vs applied V_{gs} characteristics. This rules out the possibility of the shifts being due to the readout circuit and increases the likelihood of it being due to the sample solution setup containing the reference electrode, dissolved ion and the sensor itself.

To negate the unaccounted sudden DC offset, the voltage values as input data in the training set are instead taken with respect to the response of the sensor in DIW taken prior to the measurement instead of direct values of the sensor in the ionic solution as in (5). Any variations due to noise and behavior of the reference electrode included would exist in both values hence would be negated in (5). This is done assuming that sensitivity is not affected in the DC shifts and that offsets are limited to DC voltage shifts as observed in the measurements taken:

$$V_{input\ data} = V_{sensor\ in\ solution} - V_{sensor\ in\ diw} \tag{5}$$

Table 1 and 2 compares the mean square error and correlation respectively between taking voltage directly from the interface circuit to referencing the voltage value as in (5). The comparison is done for readings between different samples, sensors, type of sensors and days of measurement. The sensors are assumed to be from the same fabrication process and membrane preparation. For same sensor/same concentration but separately prepared, Mean Square Error (MSE) is improved for all sets of comparison. For sensors demonstrating most of the shifts, repeatability is improved by an average of 98.3% for mean square error and pushing correlation to above 0.9 for every repetition. Even for sensors that did not demonstrate sudden DC shifts, the voltage variation is still improved by an order of magnitude.

Table 1: Comparing MSE between voltages taken directly to voltages referenced to DIW

Comparison	MSE direct values	MSE referenced value	MSE improvement (%)
Different samples	0.3700	0.0011	99.7
Different K ⁺ sensors	0.0044	0.0009	79.5
Different NH ₄ ⁺ sensors	0.0074	0.0005	93.2
Different days	0.2400	0.0080	96.7
Overall Average	0.1600	0.0026	98.3

Table 2: Comparing correlation between voltages taken directly to voltages referenced to DIW

Comparison	MSE direct values	MSE referenced value	MSE improvement (%)
Different samples	0.52	0.92	40.0
Different K ⁺ sensors	0.99	0.99	-
Different NH ₄ ⁺ sensors	0.98	0.98	-
Different days	0.70	0.92	22.0
Overall Average	0.80	0.95	15.5

Having removed most of the ambient noise affecting the electrical response, the sensor response is still subjected to variation inherent in the sensor structure and naturally occurring noise from the setup environment. This can be observed by the overlapping between categories in the input space as shown in Fig. 10. In this scatter plot, values of log of concentration are rounded to integer values -6 to -1 to represent 6 categories of NH₄⁺ and K⁺ sensor responses. It is found that 85% of the voltage values lie in overlapping regions. This supports the suggestion that the sensors need post-processing stage for the purpose of identifying main ion concentration in the presence of interfering ions.

Feedforward neural network: The performance of the multilayer perceptron performing regression approximation on the K⁺ concentration is shown in Table 3. The regression and MSE are based on test data on a trained network. The measured data used are voltages referenced to DIW. Regression on measured data results in 0.8065 regression factor. Network performance on artificially generated data based on (1) results in a much improved factor of 0.98. This is expected as actual measured data includes noise from background ion, device fabrication variation and noise from the environment. A minimum of 5 neurons are required in the hidden layer to achieve at least 80% correct estimation of the ion concentration. Hidden neurons exceeding 20 will terminate the training prematurely as the validation data starts to indicate over-fitting.

Table 3: K⁺ concentration approximation with feed forward neural network multilayer perceptron back-propagation learning

	Measured data	Simulated data
Epoch	42.00	20.00
Regression factor (test data)	0.81	0.98
MSE	0.15	0.02

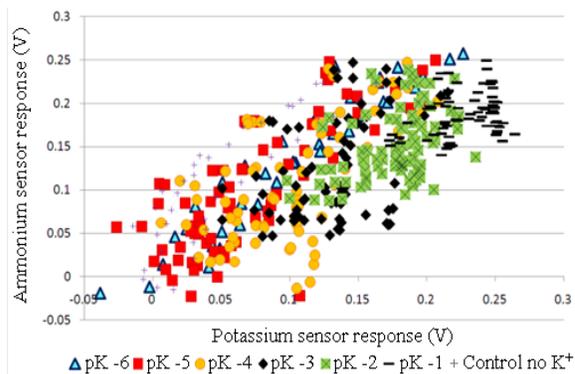


Fig. 10: Overlapping of K⁺ concentration categories -6 to -1 in the input space of K⁺ and NH₄⁺ sensor response

CONCLUSION

Knowledge representation of parallel K⁺ and NH₄⁺ ISFET sensors DC voltage response in mixed K⁺ and NH₄⁺ ionic solutions are obtained. The data set is designed to be used for a neural network supervised learning post-processing stage with the sensor voltage response as input data and the known concentration of prepared samples as target data. Data shows that the readout circuit is able to capture the ionic activity in chemical solutions by responding with indicative voltage values. Issue of repeatability of measured data is investigated and improved by referencing the output voltages to sensor response in DIW as offset voltage value prior to measurement in ionic solution to be used. Overall performance of the sensor shows heavy overlapping of data in the input space of K⁺ and NH₄⁺ two-sensor array suggesting need for a non-linear post-processing stage. The non-linear classifier MLP is able to approximate the K⁺ concentration weakly despite inherent noise in the structure.

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