Artificial Olfaction in the 21st Century

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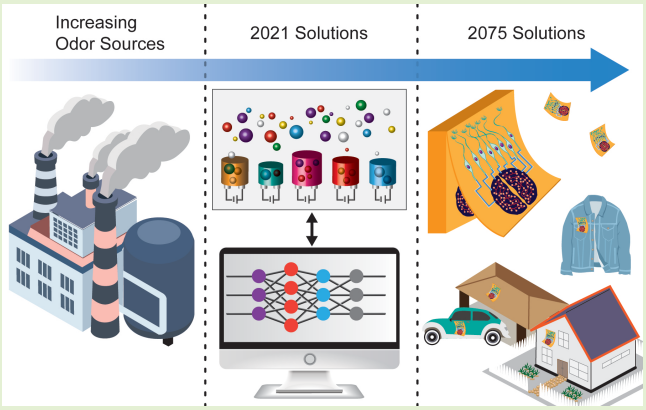
***Abstract*—The human olfactory system remains one of the most challenging biological systems to replicate. Humans use it without thinking, where it can measure offer protection from harm and bring enjoyment in equal measure. It is the system's real-time ability to detect and analyze complex odors that makes it difficult to replicate. The field of artificial olfaction has recruited and stimulated interdisciplinary research and commercial development for several applications that include malodor measurement, medical diagnostics, food and beverage quality, environment and security. Over the last century, innovative engineers and scientists have been focused on solving a range of problems associated with measurement and control of odor. The IEEE Sensors Journal has published Special Issues on olfaction in 2002 and 2012. Here we continue that coverage. In this article, we summarize early work in the 20th Century that served as the foundation upon which we have been building our odor-monitoring instrumental and measurement systems. We then examine the current state of the art that has been achieved over the last two decades as we have transitioned into the 21st Century. Much has been accomplished, but great progress is needed in sensor technology, system design, product manufacture and performance standards. In the final section, we predict levels of performance and ubiquitous applications that will be realized during in the mid to late 21st Century.**



***Index Terms*—Artificial olfaction, electronic nose, machine olfaction, odor detection, machine learning, headspace sampling, VOC analysis.**

I. Introduction

RESEARCHERS and sensor professionals have taken on the formidable challenge of accurately, repeatably, and conveniently measuring and characterizing odors that regularly occur in our indoor and outdoor environments. In the human nose (see Fig. 1), a sniff of incoming air carrying odor-generating molecules is directed via the nasal conchae onto the olfactory sensory mucosa located at the top of the nasal cavity. There the mixture of odorous molecules interacts with olfactory sensory neurons that house thousands of biomolecular receptors, which serve as odor sensors. This sensory information converges in the olfactory bulb. For each sniff, the bulb creates an outgoing response that is relayed to higher levels in the brain.

Now let us imagine humans living near a malodor generating source such as a garbage landfill as depicted in Fig. 2. Their olfactory sensory systems in Fig. 1 will be fully engaged, all-day and all-night, monitoring plumes of odor passing their way. Their higher brain levels will receive the olfactory signals and decision making will result (ignore, complain, deploy air freshener, wear a gas mask, buy a new house, etc.). The individual doing the sniffing is the odor monitoring instrument in this scenario. Let us replace that human with an electronic odor sensing device and a micro-processor/computer. Such an odor sensing device is often called an electronic nose (eNose), though it also comes with many other names, such as artificial or machine olfaction. We then have the case as shown in Fig. 2 entitled "An eNose in action."

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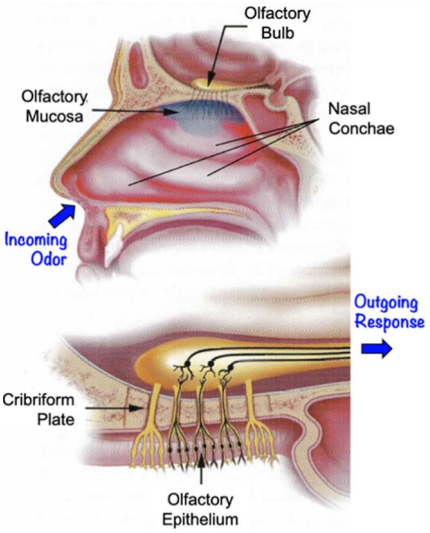

Fig. 1. Human olfactory anatomy (Adapted from [1]).

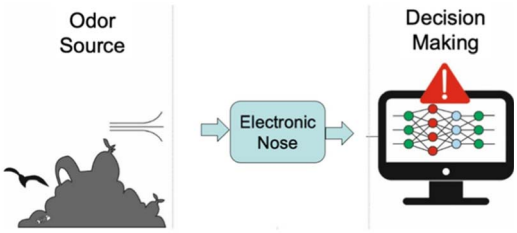

Fig. 2. An eNose in action. Example odor source from a landfill site.

The eNose system in this application should be able to operate continuously and accurately measure and document the odor challenge (in human perception terms) at that particular location. This odor monitoring system should generate specific helpful reports for the odor producer, the odor sniffer, and local regulatory authorities. Such an affordable device is not currently available for this application. Even though thousands of such devices are needed around the world, that market has not yet produced a commercially viable unit to meet this unfulfilled need.

In 2002, the IEEE Sensors Journal published its first Special Issue. Its title was Artificial Olfaction (Guest editors: H. T. Nagle, J. W. Gardner, and K. C. Persaud) [2]. Ten years later in 2012, another IEEE Sensors Journal Special Issue appeared that was entitled Machine Olfaction (Guest editors: J. W. Gardner, K. C. Persaud, P. Gouma, and R. Gutierrez-Osuna) [3]. As we approach the 20th anniversary of that first olfaction special issue, we offer this appraisal of past accomplishments and potential upcoming advances in the olfaction field. First, we provide a brief history of gas sensing technologies in the 20th Century that have served as the basis for modern developments. Then we review progress made in the early decades of the 21st Century. Those advances have produced numerous unique eNose devices for many different applications. We conclude this article with an assortment of our predictions that we envision as the 21st Century advances. We hope you enjoy it.

II. The 20TH Century

Throughout history, the presence of disagreeable odors in ambient air from poor sanitation and hygiene, tainted food, and industrial processing of materials and waste has been a concern across all cultures. Approximately a century ago, the technical challenges of measuring malodorous emissions were addressed in an article entitled "Odors and their Travel Habits" by Louis Tribus [4] published in Transactions of the American Society of Civil Engineers. Tribus discussed the difficulties of identification of offensive gases and their chemical constituents, quantifying odor dispersion and its dependence on atmospheric conditions as well as individual differences in odor perception and tolerance of persons exposed to the odors. Since that time, progress in the instrumental documentation and quantification of odors has been a slow and laborious process. The first step forward was the introduction of gas chromatography (GC), a powerful analytical chemistry technique that was first formalized by James and Martin in 1952 [5]. GC was quickly adopted to separate the individual compounds in complex odor mixtures with each detected compound appearing as a single peak in the analysis [6], [7]. The goal of GC analysis was to provide a characteristic signature for odor samples that would provide identifiable and predictable patterns. However, GC analysis has been found to have limitations for characterizing odor quality since the amplitude of the peaks is not consistent with sensory relevance. Small peaks can correspond to potent sensory compounds with low odor thresholds, and large peaks can correspond to compounds with no odor at all.

The development of compound-specific sensors for detection of targeted odorous chemicals including hydrogen sulfide (H_2S), ammonia (NH_3), and ethanol (C_2H_6O or EtOH) emerged during the decades of the 1960's and 1970's [11]–[28]. The impetus to develop H_2S sensors arose from the fact that this colorless chalcogen hydride gas (often called sour gas) has the characteristic foul odor of rotten eggs and can cause sudden death at elevated concentrations. H_2S is generated from the microbial breakdown of organic matter in anaerobic environments such sewers, swamps, landfills, and confined animal operations. It is also released during drilling activities for oil and natural gas and during volcanic eruptions. A variety of sensor principles were utilized to detect H_2S during this early historical period. Representative examples include: an electrochemical cell with a gold working electrode [8], a thin film semiconductor with film principally comprised of stannic oxide with several dopants [9], silver deposited on a thin dielectic film [10], a combined specific ion and reference electrode structure having an air gap, gas permeable membrane [11], a gas-sensing electrochemical cell comprised of a potentiometric sulfide ion sensitive electrode and a fluoride ion-sensitive electrode [12], a PbSe epitaxial film [13], a hydrogen ion selective (pH) electrode with a reference electrode and a sulfide ion selective electrode [14], and a thin film of metal oxide with a surface activated by the deposition of a catalyst [15].

Numerous commercial portable direct-reading H_2S meters as well as line-operated AC instruments were developed by the mid-1970s utilizing three different principles

of operation: solid state electrochemical, wet electrochemical, and photoionization. Thompkins [16] assessed the performance characteristics of nine prominent commercial H_2S instruments under a contract for the National Institute for Occupational Safety and Health (NIOSH) in the United States. He evaluated these instruments on thirteen performance characteristics including: response time, linearity/accuracy/precision characteristics, zero and span drifts, stability of the calibration setting, useful range of the meter, temperature effects, humidity effects, interference effects (with water vapor, CO, SO_2, and benzene), reliability information, warm-up time, accuracy of the manufacturer's calibration, noise, and recommended use technique. The results showed that the majority of these instruments had performance flaws including slow or marginal response time, negative bias for high concentrations, imprecision and deterioration of accuracy over time, large temperature and humidity effects or malfunctions during temperature and humidity protocols, interference from water vapor, CO, or SO_2, zeroing difficulties, and inability to adjust to full span response. The conclusion of this analysis was that new standards were needed for instruments designed to monitor H_2S for conformance to safety and health standards set by regulatory agencies. These standards included performance and construction characteristics that would ensure accurate H_2S concentration measurements and provide protection from exposures that might endanger health.

During the decades of the 1960's and 1970's, electrochemical sensors were also developed to detect the presence of ammonia that has a sharp, suffocating odor [17]–[19]. In nature, ammonia occurs in human sweat [20] and is generated during decomposition of plants, animals, and animal waste. The United States military utilized a device called a "people sniffer" during the Viet Nam War that could detect traces of ammonia produced in sweat to locate enemy troops hiding in the jungles [7]. During the same time period, a variety of sensor types were designed to detect ethanol that has a pungent somewhat sweet odor. Examples include perovskite oxides [21], a sintered SnO-ZnO-FeO pearl with a resistance wire [22], and an electrochemical oxidation device [23]. Other sensor technologies were also introduced during this early historical period. Odorous organic vapor from the air within reusable glass and plastic containers was sensed by a hydrogen flame detector that generated ions and gave rise to a small electric current [24]. An array of electronic olfactory detectors with an organic semiconductor barrier layer was developed by Meinhard [25]. The organic materials utilized in the detectors had electrical properties analogous to N and P conduction in inorganic semiconductors and gave characteristic responses to sulfur dioxide and certain amines. Other early devices for sensing odors included a film-coated thermistor [26], an electrolyte-metal interface to which a low DC potential was applied [27], and a polar vapor detector based on thermal modulation of contact potential [28].

Although early sensory devices were able to detect single odorous compounds, their applications were limited, and a functional artificial or machine olfaction device that incorporated design principles similar to mammalian olfaction did not emerge until the 1980's. Persaud and Dodd [29] introduced the first intelligent chemical multisensor array that could classify multiple types of odorants including mixtures of odorous volatile compounds as well as single gases. Their array consisted of three commercial gas sensors composed of metal oxide semiconductor materials with differing relative sensitivities, and the pattern or response spectrum across these sensors corresponded to the odor quality. A similar instrument based on six different semiconductor oxides was constructed several years later by Kaneyasu *et al.* [30]. Gardner and Bartlett [31] provided the first definition in the archival scientific literature for these artificial, instrument-based olfactory devices as follows: "An electronic nose is an instrument, which comprises an array of electronic chemical sensors with partial specificity and an appropriate pattern-recognition system, capable of recognizing simple or complex odours."

During the last decade of the 20th century, research into eNose technology exploded with the introduction of innovative sensor materials, methods for odor handling and delivery, and instrumentation. New strategies were generated for performing signal conditioning and preprocessing, pattern recognition and analysis, and classification of the characteristic fingerprints from the sensor array. Applications of eNose technology were introduced into domains of environmental monitoring, medical diagnostics and health monitoring, recognition of natural products, process monitoring, food and beverage quality assurance, automotive and aerospace applications, detection of explosives and cosmetics and fragrances. Dozens of companies began designing and selling eNose instruments globally to meet the demand in expanding markets for desktop-based and handheld devices. These advances were summarized in "Handbook of Machine Olfaction: Electronic Nose Technology" published in 2003 [32]. While immense progress was made in instrumental odor sensing and eNose technology by the end of the 20th century, there continue to be challenges that require substantial refinement and improvement in the performance of these instruments as we move further into the 21st century.

III. The Early 21st Century

Based on the solid foundations developed during the 20th Century, many eNose devices like the one depicted in Fig. 2 have been commercialized. In this section of this article, the functionality of these eNose units is examined using the configuration depicted in Fig. 3. Each eNose must provide the functionality of three subsystems (Sampling, Sensors, and Data Analysis). Each of these subsystems will now be summarized in more detail.

A. Sampling

Sampling is the first subsystem in an eNose system. Gas samples enter from the source and are delivered to the sensor chamber. The various stages along the way can be described as capture, transfer, conversion, and preconcentration as shown in Fig. 4. The specific application for which the eNose is designed specifies which of these stages are needed and the importance of those that are implemented. For example, an eNose instrument in which a sensor array is inserted directly into a gaseous environment will require none of these stages.

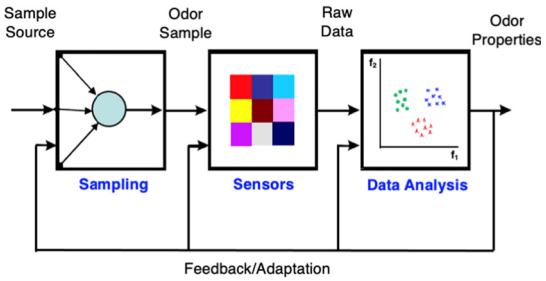

Fig. 3. eNose subsystems.

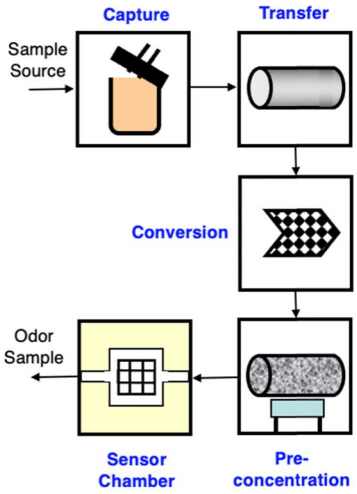

Fig. 4. Sampling.

On the other hand, all the stages of Fig. 4 will be required if the eNose employs a preconcentrating cloth patch (coated with a chemical agent for conversion to a more detectable molecular form) that is placed in a gaseous environment for several days, then capture-sealed in a plastic bag and transferred to an analysis laboratory. There the patch is removed from the plastic bag and placed in an instrument that is heated to release converted odorous molecules over a sensor array.

It is clear that an extremely good instrument can give extremely poor results if attention is not paid to sampling and the context of what is being measured. Many eNose instruments are based on sensors that transduce a physicochemical interaction that occurs when molecules adsorb onto the active surface. An odor normally comprises a complex mixture of different compounds that bind with varying affinities to the surface of the sensor. Molecules compete for adsorption based on their affinity and relative concentration and these dynamic processes are influenced by factors such as temperature and pressure. Chemical molecules also interact with each other and both physical interactions as well as chemical reactions occur, changing the nature of what is being sensed. Electronic nose devices are based on arrays of sensors that display differences in selectivity to different classes of chemical species and are not devices that separate compounds sequentially as in the case of a gas chromatograph. Hence, to obtain repeatable results requires good understanding of the matrices that are being measured, as well as carefully framing the questions that are being asked of the system. As shown in Fig. 5 the process

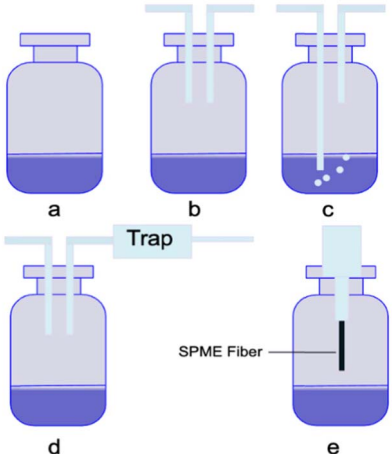

Fig. 5. Headspace sampling strategies.

of sampling involves generation of a representative headspace, transferring it to the measurement device without losing it and may optionally involve converting or modifying the sample e.g. to remove interferents or preconcentrating the sample to achieve detection at low concentrations or to selectively detect certain components.

Many of the sampling methods used for eNoses are well established from methods used for sample preparation in gas chromatography. Sampling methods for eNoses have been reviewed by Burlachenko *et al.* [33], Rouseff and Cadwallader [34], with many applications especially in food and beverage analysis [35]. The fundamental concepts can be summarized in terms of static and dynamic headspace sampling, often combined with preconcentration methods such as purge and trap or solid phase microextraction.

1) Static Headspace Sampling: Often, we wish to measure volatile compounds that are present in a liquid or solid matrix. So, for example a perfume may consist of a matrix of water, alcohol, and a mixture of essential oils. If we place a sample in a glass vial, close it and allow it to equilibrate, what appears in the headspace that is sniffed depends on the thermodynamic equilibria between all of these components and the headspace above (Fig. 5a). These thermodynamic properties dictate the distribution of molecules and is described through the partition coefficient, K.

$$K = \frac{C_S}{C_G} \tag{1}$$

where C_S is the concentration of a component in the sample phase and C_G the concentration of the component in the vapor phase when an equilibrium is achieved. Compounds with a high value for K will favor the liquid phase whereas compounds with a low K will favor the headspace phase. With an eNose we analyze the headspace phase and we need to ensure that the values of K for the analytes of interest are much lower than that of unwanted components in the sample matrix. The value of K will be dependent on both the compound and the sample matrix and it will also be strongly affected by temperature. A useful equation associated with headspace sampling is

$$C_G = \frac{C_0}{(K + \beta)} \tag{2}$$

where C_0 is the total concentration of the compound in the sample and β is the phase ratio $\frac{V_G}{V_S}$ where V is the volume of gas or sample. This equation implies that decreasing β increases the concentration of all compounds in the headspace and decreasing K, for example by increasing temperature, will increase the concentration of the compound in the headspace.

If the temperature and the sample matrix are consistent then K is constant. However, while this is true for dilute solutions, inter-molecular interactions may cause deviations at higher concentrations. In our vial there may be a variety of compounds present. Each compound vapor will contribute to the total vapor pressure observed inside the vial and the partition coefficient K is given by

$$K = \frac{p_{total}}{p_i^0 \gamma_i} \tag{3}$$

where p_i^0 is the vapor pressure of the pure compound i in the headspace vapor, that is proportional to $x_{S(i)}$ which is the mole fraction of compound i in the liquid phase and Υ is the activity coefficient which will change with concentration of the compound.

However, when we are dealing with very dilute solutions both K and Υ are essentially constants as there is little opportunity for intermolecular interactions. In this case Henry's law is applicable which states that the amount of a gas dissolved in a liquid is directly proportional to the partial pressure of that gas at equilibrium with that liquid at a constant temperature. This can be written as

$$p_i = H_i x_{S(i)} \tag{4}$$

where H is Henry's constant [36]. This is most useful for electronic nose applications especially when the sample of interest is hydrophobic in nature, favoring the build-up of high headspace concentrations from an aqueous matrix. For example, a concentration of only 0.0005 mol fraction of 2-butanol in water will provide about 236 ppm of 2-butanol in air at the air-water interface [37].

This technique has been implemented by many researchers as exemplified by [38]–[41] for quality control of hams, many liquid samples, wine ageing and others, while instruments for non-invasive measurement of water quality based on the principles of Henry's law have been commercialized by Multisensor Systems Ltd, UK for detection of traces of oil, BTEX, trihalomethanes and ammonia in an aqueous matrix (www.multisensors.co.uk).

2) Dynamic Headspace Sampling: Dynamic headspace is a technique very similar to equilibrium (static) headspace sampling but is intended to direct most of the headspace vapor across the sensor array in the eNose instrument (Fig. 5b). Two needles are used to puncture the headspace vial seal - one to introduce a carrier gas, which may be air or an inert gas, and the other to provide an outlet. This produces an exponential dilution of headspace described by:

$$C_t = C_0 exp\left(\frac{-F_p t}{V_G}\right) \tag{5}$$

where C_t is the concentration of a compound in the headspace after time t, C_0 is the initial concentration in the vial, F_p is the purge rate of the carrier gas at the temperature and pressure of the vial, t is the purge time, V_G is the headspace volume in the vial. Variations on this technique include placing the inlet tube below the liquid matrix in the vial (Fig. 5c), essentially stripping volatiles without generating an equilibrium headspace. These techniques are reviewed in detail by Wojnowski *et al.* [42].

For many applications it may be advantageous to trap the volatiles from the headspace on an absorbent bed and then subsequentially thermally desorb this directly into the inlet of the eNose (Fig. 5d). This is a method of preconcentration known as purge and trap that is often used in gas chromatography.

Dynamic headspace sampling has been optimized for many applications including drug detection [43], food volatiles [44] and has been refined to inside needle dynamic extraction (INDEX) technology to extract headspace of juice aroma volatiles [45].

3) Solid Phase Microextraction: Solid phase microextraction (SPME) [46] is another technique that can be used to extract and concentrate compounds from headspace vapor. Instead of using carrier gas to sweep or pulse the headspace vapor out of the sample, SPME essentially inserts a 'sorbent material' into the headspace vapor inside the vial. The preconcentration that occurs reflects a partition coefficient between the adsorbent and the headspace vapor and is normally implemented in the form of a coating applied to a fused silica fiber which is located within the needle of a special syringe as shown in Fig. 5e. The needle pierces the seal of a vial containing the sample and the coated fiber extends down into the headspace absorbs compounds from the vapor. After a period of equilibration, the fiber is drawn back into the syringe needle which itself is withdrawn from the vial and inserted into a heated inlet of an eNose instrument where the trapped volatiles are desorbed directly on to a gas sensor array. Apart from preconcentration, this method allows selective sampling based on the sorbent matrix used, eliminating interferents such as water vapor that plague many sensor measurements. Hence it is of widespread use in eNose technology e.g. for rancidity in oils [47], dairy products [48], potato pathogens [49], wound monitoring [50] and cancer detection from urinary volatiles [51]. Choice of the appropriate SPME fiber is often crucial to success and requires understanding of the nature of the compounds to be preconcentrated. SPME materials may be based on absorbents or adsorbents. Absorbent fibers partition analytes into a coating material. The ability of the coating to retain and release the analyte is dependent on the polarity, thickness of the coating and the size of the analyte. Thick film coatings have high sample capacity. Adsorbent type fibers extract analytes by physically interacting with the analytes. Adsorbents are generally solids that contain pores or high surface areas. There are several commercially available SPME sorbents with different polarities, such as hydrophobic (Polydimethylsiloxane (PDMS)), hydrophilic such as polyacrylate (PA) as well as bipolar materials such as Carboxen-PDMS (supplied by Supelco).

4) Complex Sampling Applications: Electronic noses are now addressing applications that previously required very

sophisticated instrumentation for analysis. One active area is the non-invasive metabolomic analysis of breath for diagnosis of several medical conditions, where compounds of interest are often found at ppb levels in a matrix of water vapor, oxygen and carbon dioxide at percentage levels [53]–[57]. It is the presence of multiple biomarkers at trace levels that allow diagnosis of a condition such as lung cancer. The lungs are in a dynamic equilibrium with the volatile compounds dissolved in the bloodstream and this reflects the sampling theory described above. As the composition of air taken from upper part of the lungs is different from deeper alveolar air this means that differential sampling of exhaled breath of a patient may be required [58]. Sampling is a critical issue in terms of the results′ quality and measurements′ success. Interference from atmospheric contaminants, patient preparation and sample collection are among the issues of concern for disease diagnosis by breath testing. Increasing multidisciplinary holistic knowledge about the matrices to be measured and interferents is driving the engineering of better sampling systems.

5) Integration of Sampling Systems With Sensors: Sampling delivers odorant molecules to the sensor technologies described in the next section. The utilization of silicon micro-machining technology and inkjet printing are now pushing the integration of micro-preconcentrators [59], separation columns, and many types of detectors (including micro thermal conductivity detectors, microflame ionization detectors, surface acoustic waves, chemiresistors and micro differential mobility spectrometers) [60], taking eNose technology into new directions for the future.

B. Sensors

Figs. 3 and 4 provide details of the capture and delivery of odorous gas sample to the sensor subsystem. Once the sample arrives, the following actions are performed as outlined in Fig. 6. Here, the first step is the initialization of the micro-controller. This then applies an excitation signal to the sensing element, through a signal conversion stage. This excitation could be the application of a voltage to the sensing or heating element (as in chemoresistive sensor) or initializing a power setting on an optical sensor. (note, the micro-controller and signal conversion stages may not be present in all odor detection systems and some sensors undertake this task internally - as in digital sensors described later). The sensing element is generally a device that converts chemical information into an electrical signal. This conversation could operate through principles including a change in conductance, capacitance, frequency, work function, optical absorbance, charge or similar approach [61]. The sensing and transduction steps are also very dependent on the sensing technology being employed. The modulation/detection, signal conditioning, and signal conversion steps are all electronic operations common in many electronic noses. The following section provides an overview of current commonly used sensor technologies deployed in electronic noses.

1) Discrete Gas Sensors: The array of gas sensors within an eNose is one of the most critical and challenging aspects of these units; it is the part that interacts directly with the

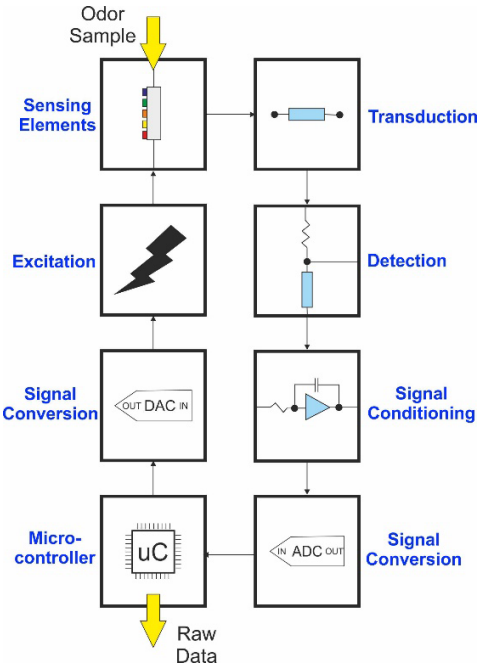

Fig. 6. Sensors.

odor. A major issue for an eNose designer is where to obtain their sensors. There are a small number of commercial gas sensor companies (a fraction of the total number of instrument manufacturers), but their sensors are often not designed specifically for eNose applications, whilst in-house gas sensors are challenging to produce reliably in large numbers, resulting in a high sensor unit cost. This has led to many eNoses using commercially available sensors, even if they are not ideal. For an eNose, these sensors are formed into arrays and must have a number of characteristics to be useable; these include diversity (that is, differences between different sensors), high sensitivity, rapid response times, a return to baseline and be tolerant of environmental conditions (e.g. changes to temperature and humidity). The first eNoses used chemoresistive metal-oxide (MOX) material gas sensors, initially 3-4, climbing to 10-18 in the larger commercial systems produced, among others, by AlphaMOS (FOX, France) and Airsense (PEN3, Germany) [62]. MOX sensors fulfilled many of the requirements for these eNoses, but often had issues surrounding diversity, the environment and drift. Most MOX sensors use SnO_2, WO_3 or ZnO in their construction, with some variance created by doping and operating temperature. They typically have high power consumption (>100mW) and operate in excess of 150^oC. Furthermore, these n-type materials are badly affected by humidity and long-term drift [63]. Most common manufacturers are Figaro and FIS gas sensors (Japan), though more recently Winsen Sensors (China) are producing sensors of similar characteristics and Alphasense (UK) producing a p-type sensor [64]–[67]. These devices have only had small improvements over the last 10 years, with a slight reduction in power consumption, some improvements in sensitivity and repeatability. The biggest advancement by far is that of MOX based gas sensors now being miniaturised and being produced on silicon based micro-hot plates. This micro-level device uses thin layers of silicon oxide with embedded heaters

to reduce the power consumption. This has meant that these sensors can be more easily integrated with modern electronics and can be packaged in standard electronic packages [68]. This is revolutionising gas sensing, as sensor systems can be battery powered, easily fitted onto PCBs and be much lower cost. Both traditional gas sensor companies (such as Figaro) are now being challenged by traditional electronic companies (such as Bosch and IDT), as well as newer companies (such as Sensirion and ScioSense). A further advancement on these has been the creation of digital gas sensors from a number of these manufacturers. The inclusion of an ASIC (Application Specific Integrated Circuit) or microcontroller with the gas sensor has allowed digital interrogation of the sensing material without the need of additional electronics. These sensors are now becoming common for indoor and automotive cabin quality applications [69]. However, unlike their analogue counterparts, for the eNose developer, the opportunity to alter the operational mode of the sensor (for example change the temperature or the way the sensor resistance is measured) is closed off. In addition, these sensors often have algorithms within the device that are not designed for eNose operation and give a faulty output to rapid changes in odor concentration. The long-term reliability of these sensors and repeatability is still questionable, but over time as these sensors improve, it is very likely that they will become more common place in eNose systems [70].

Though MOX sensors are the most commonly used in eNoses, they are not the only ones. As detailed earlier, electrochemical sensors were very common in eNoses in the early years, but now only rarely used, even though there have been great strides in repeatability, sensitivity and environmental tolerance to meet the growing demands of safety and pollution monitoring. Such sensors are now being applied in arrays to the measurement of inorganic gases, with a few examples using them as electronic noses in agricultural settings [71], [72].

A further previously common family of sensors were polymers. These were either conducting polymers (CP) or composite polymers, with the former using the material themselves as the conduction channel, whilst the latter used an additive to create a conductive path, which was then reduced with exposure to an odor through a swelling effect, with carbon being commonly used. Though researchers are continuing to work on these sensors and which are deployed in the Sensigent Cyrano and Roboscientific Electronic noses, their widespread use is still limited, and sensors are not being produced in large numbers [73]–[76]. There is some evidence of a return, as these sensors can be manufactured to be very small and can operate at room temperature. They are particularly beneficial to areas including flexible sensors, where there is the need for flexibility and low operating temperature [77].

Another common eNose gas sensor is mass measurement through quartz crystal microbalances (QCM) and similar piezoelectric devices (such as SAW devices). Instead of measuring a change in resistance or output current, these sensors operate through a change in natural resonance, where the odorant adheres to a chemical sensitive layer on the device, increasing its mass and altering its frequency. A broad range of different sensing materials has been used for mass sensors, however, common materials are conducting polymers, stationary phase materials and MOX materials (as mentioned earlier) [78]–[80]. More recently, MIPS (molecular imprinted polymer sensors) and MOFs (metal organic frameworks) have found favour with mass devices due to their greater sensitivity over more traditional polymers sensors, and still have the advantage of room temperature operation [81], [82]. In the case of MIPs the sensors are formed using a templating approach whilst the target chemical is present. Thus, after the removal of the target chemical, receptors are formed in the polymers with an affinity and sensitivity to that target molecule [83]. This makes it ideal for QCM based sensors [84]. QCMs are incredibly sensitive and have previously led to the development of commercial eNose instruments, such as the LibraNose (Italy) [72], [60]. However, integration of the sensor with electronics remains challenging, partly due to temperature intolerance.

There are also families of sensors based on carbon nanomaterials and more recently graphene. Large sums of research funds have been spent on these devices – potentially more than the total value of the gas sensor market. They offer high-diversity and high sensitivity with small form factor [74], [85]. There have been a number of start-ups in the field, (such as AlphaSenzor, Aernos and C2Sensor), but challenges of repeatability, reliability and market access are limiting their widespread use.

However, many researchers have produced electronic nose systems using these carbon-based sensors. In this case, they have both manufactured the array and then applied to an application. These sensors are formed from either single-wall or multi-wall carbon nanotubes that are configured in a chemiresistor or ChemFET (where the CNT forms the channel of the transistor). The CNT can be used pristine, used singularly or in bundles, but they have limited selectivity and so are commonly doped with, for example, a noble metal (such as Pd), added to a polymer (working on a swelling principle) or a metal-oxide [86]–[88]. These sensors can then be created into sensor arrays to operate as an electronic nose [89]–[91]. An alternative carbon-based material is graphene and reduced graphene oxide. These 2D materials can be operated in a chemiresistor, chemFET or chem-capacitor, similarly to CNTs and formed into various shapes with sheets or ribbons being common [92]. The materials are often doped to improve selectivity with noble metals, polymers and metal-oxides again being common choice material [93]–[95]. A number of researchers have also produced electronic nose instruments by creating arrays of graphene sensors for a number of applications [96], [97]. Another common nanosensing material is the combination of metal nanoparticles (e.g. Ag or Au) and a polymer to form a chemiresistive sensor. The metallic particle is formed into a monolayer with an organic film component. The VOC either expands or contracts the polymer film causing a reduction or increase in resistance [98]. These sensors have been formed into a number of sensor arrays for a range of electronic nose applications [99], [100]. These are only exemplars of the broad range of nanosensors in development and it is an open question on how successful these sensors will be, but they may provide

new opportunities for eNoses to become smaller, cheaper and widespread in our consumer lives.

A parallel sensor development in eNoses has been the use of optical dyes as chemical sensors. These devices produce a colour change when exposed to an odor (chemical response), which can be measured using a modern CCD or CMOS camera [101], [102]. The most common materials are based on Lewis acid/base dyes, Brønsted acidic or basic dyes and dyes with large permanent dipoles (i.e., zwitterionic solvatochromic dyes), though other dyes are available. These materials can be printed onto paper or plastic to create ultra-low-cost gas sensors. In use, images are taken before and after exposure to create a differential image. Such sensors can be made to be reproducible or single use, depending on the need [103]. An alternative technique based on an optical approach is surface plasmon resonance (SPR). Here a cross-sensitive micro-array of sensors is interrogated using an optical light source (usually an LED) and detector. The system measures the absorbance of an odor onto a metallic or organic molecule, though metal oxides and nanomaterials are commonly used [104]. These molecules are deposited onto a gold coated prism and the response measured as a function of the reflectivity [105]. These sensors have been shown to be able to detect a broad range of VOCs and offer real opportunities for portable eNose systems, for example the NeOse Pro (Aryballe, France) [106].

2) Tuneable Gas Sensors: A traditionalist approach to eNoses is the need to use a sensor array in its construction. However, it is now being accepted that using a single sensor and sweeping its characteristics to create an array of "virtual" sensors is equally as valid. In this case, we have defined tuneable as the application of a method that alters the sensitivity or specificity of a sensor to chemical groups. This may not be as simple as changing the magnitude of the response, but could also be an alteration of response and recovery times. Though it may seem a better approach to use a large array of different discrete sensors, the diversity in commercial sensors is relatively low and gaining more information out of a single sensor is a relatively cheap. Previous to this, the only approach possible to increase diversity was to make large arrays of sensors (either MOX or polymers). These arrays are not the typical 6 to 32, but had more than 100 elements. For polymers this was done by using different materials, which is time consuming and difficult to construct [107]. However, for MOX sensors the same can be achieved by having a large array of sensors at different temperatures (such as the Kamina Nose). Though successful, it didn't achieve commercial success and the power consumption was considerable ($>$4W) [108]. With the creation of micro-hot plate sensors, the same could now be achieved with a smaller number of sensors that can be thermally modulated. The low time constants (sub second) means that the sensor temperature can be swept whilst in the presence of an odor producing a much higher dimensional information. This technique is used in a number of commercial electronic noses, with the AeoNose (eNose company, Netherlands) being a good example [109]. This is useful as the sensitivity and specificity of MOX sensors is a function of operating temperature [110], [111]. The same approach can also be applied to polymer sensors, using micro-hot plates to thermally modulate sensor temperature as a means of single chemical identification [112]. The advantage here is that the operating temperatures are much lower than MOX materials. This is now a common approach for many eNoses [113]. A similar approach is to measure the sensor resistance using an impedance AC approach, rather than the traditional DC technique. The AC approach provides additional information as MOX films have trapped charge on the surface of the material film, thus both the real and imaginary parts of the impedance measurement hold useful information [114]. Further work in this area has recently been found to make the MOX response linear, increase the sensitivity range and the reduce the effects of changes in humidity and environmental temperature, making it an interesting method for eNoses [115].

A more recent variant is to use UV light to modulate the sensor surface [116]. By changing the frequency, amplitude and duration of the UV exposure, more information can be gained from the MOX sensor, again producing multi-dimensional data. Furthermore, it has the advantage of decreasing response and recovery times and helping to stabilise the sensor before use.

3) Physical Sensor Measurement: For higher-end/more-expensive eNose systems, many researchers have moved away from the traditional chemical based sensing model to develop approaches that measure physical properties of molecules. Such systems offer more stability and repeatability as physical measurements are less prone to drift compared with chemical reactions. The most successful type has been the development of Ion Mobility Spectrometers (IMS) deployed as eNoses. With such instruments, it is often difficult to identify specific molecules and thus pattern-based techniques have been applied. They operate by ionising odors and then measuring the resultant ions in a high electric field. Most common types are based either on a drift tube, where the time an ion takes to traverse a tube against a buffer gas is measured, or using FAIMS (field asymmetric IMS), where the movement of ions in high and low electric fields are measured [117]. These instruments can operate close to room pressure using air as the carrier. Example instruments include Lonestar (Owlstone, UK) and ChemProX (Environics, Finland) [118], [119]. They also offer incredible sensitivity (down to parts per trillion) for some chemical families. However, they are often not tolerant to higher humidity levels and require high voltages, making them challenging to use in certain applications. A similar type of product is based on optical detection. It is well known that certain molecules absorb various frequencies of light (mostly in the infra-red) and that these changes can be measured. For odor analysis, either non-dispersive approaches (using a wide spectrum light source and optical filters) or laser scanning approaches (common detector with tuneable laser) are not common for eNoses as they are often used for single chemical detection [120]. This is because most uses of these tools are focussed on single gas detection applications, though a large range of optical designs are available [121]–[123]. Other options include optical frequency comb spectroscopy and laser based photoacoustic gas sensors.

The first one allows a broad range of optical frequencies to be measured but has lower sensitivity than fixed frequency systems and are produced by manufacturers such as Protea [124]. Photoacoustic approaches use a laser to excite a molecule which is detected by a microphone. However, it is also possible to achieve the same result using a wide-band light source and optical filters. These devices are often used for detection of small molecules including ethylene and carbon dioxide [125].

4) Biological Odorant-Related Proteins: Immobilized odorant-related proteins can also serve as sensors in eNose applications, but practical implementation of this transducer type is still in its infancy. Two types of odorant-associated proteins have been incorporated into biosensors for a variety of applications. These include: 1) odorant receptors (ORs) that are expressed in vivo on olfactory receptor neurons and relay qualitative information about odorous ligands to the brain, and 2) odorant-binding proteins (OBPs) that capture odorants in the nasal mucous of vertebrates and transport the odorants to the ORs. Approximately 400 functional types of ORs have been identified in humans [1]. Multiple receptor types are typically stimulated by a single odorant, and the patterns of activation across the spectrum of receptors allow humans to distinguish more than one trillion structurally diverse odorous molecules. Sensors consisting of five fractionated ORs from bullfrogs and one referenced phospholipid probe were able to respond to multiple odorant types in a piezoelectric biosensor [126]. Carbon nanotube field effect transistors have been successfully functionalized with ORs to detect numerous odorous analytes [127]–[129]. A carbon nanotube transistor biosensor that incorporated a canine OR was used to detect hexanal, an indicator of the oxidation of food [130]. Arrays comprised of human ORs expressed on neurons in cell culture medium are in development as eNose devices [131]. OBPs from pig immobilized on quartz crystals responded to pyrazines as well as pyridine [132], [133]. A simple multisensor array based on bovine OBPs also showed responses to odorants [134]. A water-gated field effect transistor using a pig OBP successfully detected chiral molecules of carvone [135]. Arrays have now been developed using major urinary proteins of similar structure to OBPs on diamond-based resonators [136] and have been applied to detection of traces of explosives and drugs [137]. Devices that incorporated ORs detected odorants at lower concentrations that those that utilized OBPs. Use of biological ORs in a sensor array has the potential for greater sensitivity and discrimination than non-biological gas sensors but further development is necessary.

5) Combined Systems: As stated earlier, diversity within the sensor array remains a challenge with eNoses. A further solution to this has been to combine different sensing approaches into the same unit to offer orthogonal detection and provide more information on the sample under test. The most common of these approaches has been to use gas chromatography (GC) as a means of separating out complex mixtures of chemicals and then exposing these single chemicals to a more traditional detector. For eNoses, it is not critical that the GC provides full separation, more than it enriches the information content available. The two best known commercial instruments are the zNose (Electronic Sensor Technology, US) and Hercules (AlphaMOS, France) [138], [139]. The former uses a short column and a plain surface acoustic wave (SAW) pick-up, while the latter is a two column GC with standard detectors. Both instruments operate with helium and therefore, may not be considered traditional eNoses, but do create arrays of "virtual" sensors based on temporal output, rather than spatial responses. Instruments that more closely mimic an eNose are devices such as the FIS SGC (Sensor Gas Chromatograph) that operate a GC column followed by the MOX sensor [140]. By changing the MOX sensor and the GC column, a high level of selectivity can be acquired. There are research level developments using MOX arrays as the detector to increase the diversity, but they have yet to be commonplace. Such techniques can also be applied to those tuneable instruments and units that operate by GC-IMS. With a pre-separation and a highly tuneable detector, these units provide excellent performance [141]. However, all of these instruments are large and bulky driving the need to produce micro-GCs. This has been an active area of research for almost 30 years, but there are very few commercial systems. One example is the Frog from Defiant Industries, which combines a micro-GC with a pre-concentration and a PID (photo ionisation detector) pick-up, though similar systems exist at the research level [142], [143]. Table I provides a simple comparison of some of the more common eNose sensor technologies and Table II of alternative approaches (note: we are defining GC-Sensor as a GC column as a pre-separation stage. In this case the sensor may be MOX, QCM, polymer or IMS based).

TABLE I
COMPARISON OF COMMON ENOSE DISCRETE SENSOR TECHNOLOGIES

Technology	CP	MOX	QCM	Nano
Sensitivity	Low	Average	High	Average
Selectivity	Low	Average	Average	Low
Portability	Good	Good	Good	Good
Cost	Low	Low	Low	Low
Trained Personnel	No	No	No	No
Sample Throughput	High	High	High	High
Speed	Real-Time	Real-Time	Real-Time	Real-Time
Pattern Recognition	Yes	Yes	Yes	Yes
Chemical Insight	No	No	No	No
Sensor Drift	Yes	Yes	Yes	Yes

6) Electronic Advancements: One of the many improvements in eNoses has been the electronics within them. Driven by the rise in consumer products and IoT applications, there has been rapid development in electronics, with a reduction on power and size, whilst at the same time improvements in performance [144]. For traditional gas sensors, these advancements have not been so significant as sensor voltages and power levels remain high. However, as sensors have been miniaturised and drive voltages have reduced, then sensor

TABLE II
COMPARISON OF ALTERNATIVE ENOSE TECHNOLOGIES

Technology	IMS	Optical	GC-Sensor	GCMS
Sensitivity	High	Average	Average	High
Selectivity	Low	Good	Good	High
Portability	Good	Average	Average	Poor
Cost	Medium	High	Medium	High
Trained Personnel	No	Yes	Yes	Yes
Sample Throughput	Medium	Medium	Medium	Low
Speed	Real-Time/ Offline	Real-time Offline	Offline	Offline
Pattern Recognition	Yes	Yes	Yes	Yes
Chemical Insight	Yes	Yes	Yes	Yes
Sensor Drift	Minor	Minor	Minor	Minor

interrogation becomes easier. In analogue electronics, there have been significant advances in passive components, with a reduction in size and much higher component accuracies. In active components, op-amps are now commonly operating at low voltages down to 1.2V, with rail-to-rail input and output characteristics, ultra-low power, high input impedances and zero drift. Similar improvements can be found in comparators, power systems, transistors and nearly all other common electronic components [145].

For digital electronics, with the advancements in analog-to-digital converter (ADC) resolution and cost – going from a 8 or 12-bit world to 18, 24 or even 31 bits and high-resolution, digital-to-analog converters (DACs) now common place, more granularity can be gained from sensors. In addition, with the advancements in advanced reduced-instruction-set-computing machine (ARM) processors, micro-controllers are now hugely powerful and at a cost of cents. These units are more powerful than computer based eNoses just 10 years ago. This combination has also created system on a chip (SoC) solutions that integrated analogue and digital components onto a single device, where the sensor can be biased and the responses measured. This has been applied to a range of different sensor solutions, including polymers, MOX and QCMs [60], [146], [147]. Beyond the integrated digital solutions, mainstream electronic manufacturers are now producing SoC devices for widespread use by eNose designers [148], [149].

Furthermore, the drive for communication and the use of cloud processing may bring significant changes to the use of eNoses. Wi-fi, Bluetooth® and now LoRa® have started to be seen in eNose systems and cloud computing of eNose data is likely be common soon. Current uses of distributed, low-cost air quality monitors are already using the internet for this purpose [150].

C. Data Analysis: Signal Processing & Machine Learning

Pattern recognition and by extension, signal and data processing has been considered a key element of artificial olfaction [31]. At this point it is worth noting that eNoses are not the only instrumental platform characterized by the importance of data processing. This characteristic is shared by chemical instrumentation such as NIR near infrared spectrometry [151], [152], or ion mobility spectroscopy (IMS) [153]. In the last decade, the strong stimulus received by the analysis of omics data, has consolidated the role of statistical data processing and machine learning for chemical instrumentation in areas such as metabolomics [155] and foodomics [156].

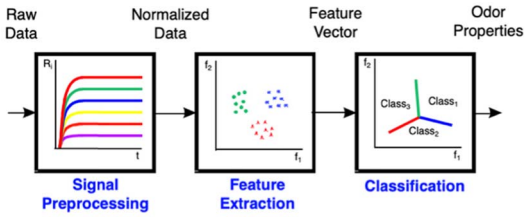

Fig. 7. Data analysis.

In its most basic architecture, the main building blocks or artificial olfactory systems from the signal and data processing point of view are displayed in Fig. 7. Raw sensors data require some signal preprocessing for bandwidth control, and basic feature extraction. Due to feature correlation, data can be projected to lower dimensionality spaces where classifiers or regressors are finally built. Many different versions and modifications of this basic scheme have been proposed in the literature for the last 20 years. However, in this last decade the contributions in signal and data processing go well beyond these individual blocks and try to face relevant problems of chemical sensor arrays. The state of the art in data processing for artificial olfaction has been reviewed on different occasions [157], [158] in this journal. In this section we are going to present novel and differential elements with respect to these previous reviews. For the sake of brevity the readers are referred to the original publications for technical details.

This last decade has meant substantial progress in the methodological maturity in the research area of signal processing as well as predictive model building and validation with an important emphasis on algorithms that improve the robustness of eNoses [159], [160]. In the following, we highlight several avenues of research that have taken an important leap forward.

1) Calibration Transfer and Datashift Correction: The instrumental stability of eNoses has been a constant concern in the community and a barrier to commercial adoption of the technology. Electronic noses often require a high number of calibration points [161], making frequent recalibration not feasible either for practical reasons or for reasons of economic cost. This limitation motivated the development of automatic and unsupervised drift correction algorithms two decades ago [162], [163]. However, the use of these algorithms was limited to research and academic settings. On the other hand, there has been increasing interest in the calibration transfer and datashift correction algorithms. These algorithms allow an update of the calibration models with a minimum number of additional calibration points instead of a complete recalibration [164]–[168]. Alternative strategies to improve sensors stability have been based on semi-supervised learning algorithms that take advantage of the availability of unlabeled samples [169]. Adaptive machine learning calibration methods

are still the object of new developments, as for instance those based on the Artificial Immune System [170].

Along the same lines, there is great interest in calibration models that can be transferred between identical instruments at the construction level. It is known that due to tolerances in sensor devices, the same model is not usually directly transferable from one instrument to another. It is in this context that transfer techniques allow full calibration of the primary instrument only and easy transfer with few points to secondary instruments [171]–[173].

Recent works have proposed methodologies for the complete elimination of transfer points by selecting predictive models that can reject instrumental variability providing instrument specific predictive models without individual calibration [174].

2) Fault Detection, Identification and Recovery: Unless careful algorithmic design is considered, poisoning events or in general sensor faults can make prediction models obsolete. Fault identification and correction has been a topic of research in the community for the last 20 years [175]. In this area, a self-repairing algorithm has been proposed based on an unsupervised online selection of reliable features [176], [177]. An alternative proposal from the same group relies on cooperative classifiers that operate in a subset of the sensor array, in such a way, that by consensus analysis it is possible to determine if some classifiers have to be discarded or added to the pool in order to have a stable performance [178]. A similar approach using SVM with kernels on subsets of sensors demonstrated the robustness of this approach towards sensors failure [179].

3) Methodological Improvements in the Construction and Validation of Predictive Models: A fundamental barrier to the commercial adoption of eNoses has been the difficult industrialization of apparently positive results in the laboratory, for their use in practical conditions in industrial, clinical, or environmental scenarios. In this sense, several studies have emphasized the importance of rejecting potential confounding factors (a deeply rooted concept from the omics sciences and epidemiology) that could be behind an apparent classification success that was not generalizable in less controlled working conditions [180], [181]. A clear example is the importance of a random ordering in the presentation of calibration samples, the use of quality control samples and blanks. Considering that eNoses are time-varying instruments, the sometimes-overlooked main confounder is the instrument drift itself during calibration, either due to environmental factors or memory effects in the system. That is why, in addition to a good experimental design in the calibration, great emphasis is placed on controlling the rigor of the validation, avoiding the pervasive but limited in scope intra-study validations. More and more studies are being found that propose validations that allow verifying instrumental robustness to small methodological, instrumental, or relative variations to the samples under study [182], [183].

4) Figures of Merit of the Calibration Model: A mature treatment of eNoses as measurement and monitoring instruments goes through an adequate definition and calculation of figures of merit that allows the objective comparison of various instruments. These figures of merit are well established for univariate sensors, but their definition and widespread use for chemical sensor arrays have been hampered by aspects such as the non-linearity of the sensors, the use of multivariate tools based on machine learning or the difficulties associated with the measured magnitude, when it is it is expressed in terms of human perception. In the last decade we have seen new proposals for the definition and estimation of important figures of merit such as selectivity [184], limit of detection [185]–[187] or chemical resolution [188]. Many of these new proposals are based on prior art [189], [190].

As mentioned previously, the characterization of the performance of the instruments goes through their comparison with human perceptions, be they qualitative or quantitative. A paradigmatic case with important environmental implications is the use of eNoses to estimate the intensity of the odor. In this case, the variability of human perception is controlled using a panel and by the use of standardized methodologies based on dynamic olfactometry [191]. In any case, instrumental validation cannot assume that the reference technique has a quasi-zero error, and alternative methodologies based on model comparison must be used, techniques that are otherwise common in the domain of biomedical engineering. Among them we can find the classic Bland-Altman method [192], [193], where the main idea is whether the objective determination can differ from the subjective one above a predefined limit dependent on the application. We speak then of the calculation of the limit of acceptance (LoA) of the differences. An alternative treatment is based on the use of regression methods that accept errors in both axes, such as Deming Regression or Passing-Bablok Regression [194].

5) Chemical Mapping and Chemical Source Localization: In recent years we have seen a renewed interest in the use of eNoses to estimate a map of the odor distribution and eventually determine its source. Substantial algorithmic developments have been proposed in the last decade on the best ways to build those maps. These algorithmic developments slightly differ in ambition and scope, whether the eNoses are mounted in mobile platforms based on terrestrial or aerial robots [195]–[199], or by deploying chemical sensor networks over the area of interest [200]–[202]. The use of machine learning methods for sensor array calibration in this application scenario has been widely explored [203]. This application must face additional difficulties such as the turbulent nature of atmospheric transport, which can be further complicated by the presence of obstacles, and changes in the direction and intensity of the wind. This gives rise to signals with a chaotic character, with rapid variations, particularly near the point of emission and even characterized by periods of intermittence [204]–[206].

In this sense, various groups have made algorithmic proposals to determine which is the best extraction of characteristics to obtain information from the chaotic chemical signal that excites the sensor elements. To the traditional use of the instantaneous value of the signal has been added the use of variance, or more complicated or bio-inspired methods [207]–[209].

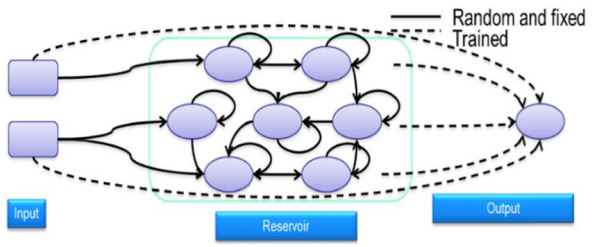

Fig. 8. Reservoir computers, a kind of recurrent neural network, has been proposed with success to improve the dynamics of eNoses subject to fast transients in continuous monitoring. Adapted from [220].

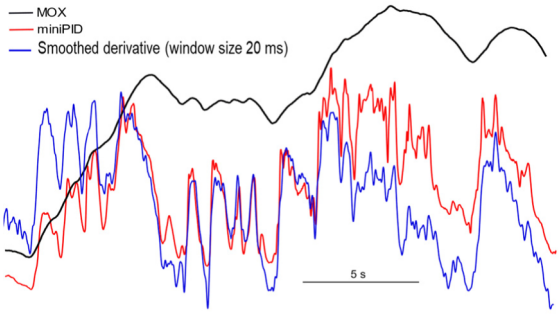

Fig. 9. Linear inverse filters may improve chemical sensors time response one order of magnitude, opening new possibilities for their use in robotic platforms. Adapted from [209].

These readings obtained at various points in time and space must be fused with some underlying probabilistic model to reduce the variance of the map [210], [211]. This underlying probabilistic map can be created using interpolation and smoothing methods [212] based on convolution kernels [213] or on physical models of dispersion [214], [215]. When the objective is only to obtain the position of the source, Bayesian inference methods can be used from the detection of events or the readings of the signal itself [216], [217].

6) Improving Sensor Array Dynamics: Electronic noses can have especially slow response times on the order of tens of seconds or even minutes depending on the instrument setup [218]. These response times pale in comparison to the dynamic response of insect antennas that can reach bandwidths of 100 Hz [219]. This slow response makes it difficult to use eNoses for applications in robotics or even in continuous monitoring in the presence of variations. rapid concentration. Dynamic behavior is very complex since non-linearities are often observed not only in static behavior, but also in dynamic behavior. That is why predictive models based on dynamic neural networks [220], [221] (see Fig. 8), or extended Kalman filters have often been used with good results [222]. It has been shown however that, if the objective is only the detection of events, the use of linear inverse digital filters is sufficient to improve response times by approximately one order of magnitude [209] (see Fig. 9).

7) Bioinspired Algorithms: The biological olfactory system of both vertebrates and insects in its information processor aspect has always been a source of inspiration in the community [223], [224]. This line of activity, continues to be active, particularly in the context of increasing interest in neuromorphic computing [225], [226]. The olfactory system performs a multitude of computational functions. Beyond a poor estimate of odor intensity, smell demonstrates excellent capabilities for detection, rejection of background noise, odor discrimination, contrast enhancement, gain control, combinatorial coding, pattern completion, and error correction [227]. Various groups have implemented bioinspired algorithms, in both insects and vertebrates, with different levels of abstraction [228], [229] and their operation has even been demonstrated in real time on a mobile robotic platforms [230]. In this sense, a recent highlight is the implementation of a bioinspired algorithm for chemical sensors on the *Loihi* neuromorphic chip by Intel [231].

The comparison between the combinatorial coding capacity between olfactory receptors at the level of the olfactive epithelium or in their response integrated by glomeruli at the level of the olfactory bulb and MOX sensors has also been the object of analysis. The results show that for the dataset studied the biological sensors are less correlated than the artificial sensors and more sensitive, which gives biological receptors greater molecular range of coverage. While limited in scope, this type of analysis sheds light on why artificial olfaction shows a limited chemical resolution compared to their biological counterparts [232], [233].

8) Data and Computational Resources Availability: Beyond algorithmic advances and changes in research focus, the last decade has witnessed a major change that improves the reproducibility of research in the field of signal and data processing. It is increasingly common for researchers to share the data sets under study and sometimes even the computational tools developed for their analysis. Data repositories such as the UCI machine learning repository [234] allow researchers to access previously analyzed data sets, reproduce the results and propose algorithmic improvements and compare them in homogeneous terms. It is expected that this trend will be completed with the possibility of sharing also the tools developed via repositories such as CRAN in R [235] and the Python Package Index [236], or directly through github [237].

D. Applications

In the past 20 years, the application of electronic nose technology has accelerated in many sectors including environmental quality of indoor and outdoor air, medical diagnostics and treatment, food quality and contamination, agriculture, forensics, and safety. Representative recent applications reported in the scientific literature are given in Table III [1], [238]. Although significant progress has been made in the performance of eNoses for these applications, challenges remain in obtaining accurate, consistent, and reproducible results over time. In 1976, Thompkins [16] noted sensor sensitivity, selectivity, nonlinearity, drift, and stability as challenges for that era's hydrogen sulfide sensors. These challenges remain for today's eNose devices. These issues must be solved as we move forward in time.

E. Standards

During the first two decades of this 21st Century, odor monitoring standards have garnered attention from academic researchers who measure odors, government regulators who specify exposure limits, and industrial facilities that release odorous gas plumes. Since international standards have proven to be very valuable in many technology sectors, the stakeholders in industries that generate, and control, odor emissions

TABLE III
RECENT APPLICATIONS USING AN ENOSE [1], [238]

Area	Application
Environmental: air and water quality	Landfills
	Oil and gas plant emissions
	Dangerous environments (e.g. explosives)
	Swine confinement facilities
	Automobile cabin air and parts
	Industrial gases
	Indoor air in buildings
	Molds growing on building materials
	Contamination of potable/tap water
	Pathogenic microorganisms in waste water
	Microbial content in rivers
Medical/ healthcare	Lung cancer diagnosis from breath test
	Head and neck cancer
	Colon cancer from breath analysis
	Urinary tract infection
	End stage renal disease
	Bile acid diarrhea
	Chronic rhinosinusitis
	Tuberculosis
	Chagas disease
	Asthma
	Cystic fibrosis
	Anesthetic dose level and identification
	Cell culture monitoring
	Stem cell differentiation steps
	Cannabis and tobacco use
Foods and Beverages (discrimination, quality, contamination, adulteration)	Distilled liquors including Scotch whiskies
	Beer production
	Rice wine
	Tea
	Coffee
	Chocolate
	Red meat, pork, and chicken
	Fish and shellfish
	Oils including olive and rapeseed
	Vinegar
	Vegetable soup
	Cheese ripening
	Food and beverage packaging
Agriculture and Forestry	Fruit maturity and quality
	Potato rot
	Tobacco
	Wood and paper processing
	Classification of plants and animals
Product quality, contamination, and forensics	Illegal drugs
	Cutting agents in cocaine samples
	Hydraulic oil fingerprint contamination on aircraft
Military; safety	Vapor analysis of explosives

TABLE IV
SUMMARY OF ACTIVE ODOR MONITORING STANDARDS WORK GROUPS (WGS)

Designator	Region/ Year	Title	Ref.
CEN TC/264 WG41	Europe (2015- pres.)	Emissions and ambient air – Instrumental odour monitoring	[1], [239]
VDI 3518-3	Germany (2018)	Multigas sensors - Odour-related measurements with electronic noses and their testing	[240]
UNI 1605848	Italy (2019)	Emissions and air quality - Determination of odor through IOMs (Instrumental Odor Monitoring Systems) and their qualification	[239]
IEEE P2520.1	International (2020-pres.)	Standard for Baseline Performance for Odor Analysis Devices and Systems	[241]
IEEE P2520.2.1	International (2020-pres.)	Standard for Machine Olfaction Devices and Systems Used for General Outdoor Odor Monitoring	[242]
IEEE P2520.2.2	International (2020-pres.)	Standard for Landfill Odor Monitoring Devices and Systems	[243]
IEEE P2520.3.1	International (2020-pres.)	Standard for Machine Olfaction Devices and Systems Used in General Indoor Odor Monitoring	[244]
IEEE P2520.4.1	International (2020-pres)	Standard for Performance of Machine Olfaction Devices and Systems for Chemical Manufacture	[245]

are turning to standards to aid their goals for developing functional and reliable products for the global marketplace. Standards set performance criteria that guide the design and manufacture products and optimize their reliability and safety. For products that are monitoring odor exposure of human beings, the perception of that odor has great influence on the impacted people's wellbeing. Therefore, important parameters to be measured, reported, and archived include the perceived odor intensity, irritation, and pleasantness. To date, several odor standard-writing groups have been established, but published standards are just emerging. Activities in Europe began about 20 years ago [239]. Recently, the IEEE Standards Association (IEEE-SA) established a collaboration with the International Society for Olfaction and Chemical Sensing (ISOCS) to develop a series of standards for machine olfaction devices and systems. Table IV summarizes several eNose-oriented standards activities from the European Union and the IEEE Standards Association. Individuals who are interested in participating in the IEEE-SA Working Groups can inquire via the references in Table IV.

IV. THE REST OF THE 21ST CENTURY

Though progress has been made during the last 20 years, much more was predicted by experts in the field. For example, Stetter and Penrose (2002) [246] gave some predictions, but no specific timeline. They foresaw improved chemical sensors and better drift correction. They envisioned chemical sensors that mimicked nature by being self-amplifying and regenerating. They suggested that chemical sensors would measure fundamental properties like solubility and binding constants. They believed that sensor arrays would compensate for patterns and sensor heterogeneity and would integrate into microfluidic systems with sampling and separation systems. They anticipated that sensor arrays would be spatially and temporally separated as is the human olfactory system. Finally, they expected pattern classification to improve. Some of their goals were achieved, and some were not. From this point forward, the future of artificial olfaction will rest on significant technical improvements that are forthcoming. These improvements include hybrid biophysical sensor technologies to capture the odor samples, ubiquitous distributed computing nodes at the edge of the

Cloud to manage and archive odor-sample data, and AI and machine-learning algorithms to make sense of massive aggregations of real-time and archived odor data. Due the recent exponential increase in processor computational speeds, reductions in electrical component power consumption, and Cloud storage capacity that seems infinite, we believe that tremendous progress in artificial olfaction is before us. Here we give our predictions. We leave it to the researchers to decide how best to get there. Please give us a grade in 25 years!

A. Predictions for 2025

1) Sampling 2025:

- Adsorbent materials with highly selective properties – Nanoparticle based materials carbon nanotubes, graphene, 2D layered materials such as metal chalcogenides, transition metal oxides, emerging materials such as layered transition metal dichalcogenides.
- Microfluidic technologies integrating sampling with sensing
- Innovative additive manufacturing utilizing printable materials for sampling, and preconcentration
- Distributed sampling and sensing for the environment.

2) Sensors 2025:

- Traditional gas sensors will remain the same, due to the conservative nature of the gas sensors industry, with incremental changes in sensitivity and specificity.
- High-end (>$20k) eNose market will move further towards physical measurement systems, such as IMS and optical measurement, and further away from traditional sensor array based eNoses.
- These high-end systems will provide significantly more chemical information on an odor and will facilitate improvements in data analysis
- Further material integration into modern electronics, with thin film MOX materials being the most common. This will facilitate wide-spread use of these devices
- Reduction in power of commercial gas sensors – achieved through a combination of size reduction, thin films and the development of materials able to operate closer to room temperature.
- Continual development of 2D, graphene, gold nanoparticle sensors. Mostly at the research level with further small start-up companies being created.

3) Data Analysis 2025:

- Algorithmic solutions for chemical mapping and odor source localization will be tested and demonstrated in real scenarios, when mounted in terrestrial and aerial unmanned vehicles. Algorithms will be sufficiently robust to wind variations in direction and intensity, as well to the perturbations due to vehicle motion.
- Commercial eNoses and chemical sensing systems will include by default auto diagnostic capabilities, informing the user about drifts and sensor faults. The faulty unit will be identified, and their signal automatically corrected.
- eNoses deployed in smart cities will provide high spatial and temporal resolution data that will be fused with environmental dispersion models.
- eNoses will self-calibrate when collocated with mobile reference environmental monitoring stations.
- Full standard pre-processing and machine learning workflows will appear for Ion Mobility Spectrometry data, enabling the use of the instruments for food quality control and food fraud detection. These workflows will include fully automated signal processing including smoothing, peak detection, peak alignment, signal deconvolution followed by predictive machine learning without any human supervision. Systems will be used without expert knowledge in data processing.
- Smart eNoses with embedded signal processing will be trained to differentiate hedonic tone in very limited application scenarios but of commercial relevance such as of malodors in home appliances
- eNoses will have subsecond response time by the combination of proper system design (sensors and fluidics) and signal processing based in inverse filters.
- Further progress will be observed in the use of insect antennae and electrode recordings of the antennal lobe neural circuitry to control demonstration robots.

4) Applications 2025:

- Several personal-use eNose devices will have been conceived, each with a large potential market. Startup companies to commercialize those devices will be operating with the support of a stable group of investors.
- More medically targeted devices will be available with regulatory approvals.
- Automobile manufacturers will be including eNose devices in the interior of some of their high-end models.
- Rental car agencies and demand-ride services will begin including eNose devices in their vehicles to promote driver and passenger safety.
- eNose devices will begin to appear in high-security areas to detect intruders, drugs, and other contraband.
- Large numbers of home safety and fire alarms will be offered with eNose device options.
- Some smartphone companies will be investigating eNose sensors for their high-end devices, other options will be available for beta testing.

5) Standards 2025:

- European standards will continue to develop. One or two will have been published and be in wide use.
- Several new IEEE odor-monitoring Working Groups will have been initiated, bringing the total to more than a dozen.
- Three to five IEEE odor monitoring standards will have been published with two or three gaining commercial adoption.
- IEEE Conformity Assessment Program (ICAP) sites are being organized.

B. Forecast for 2050

1) Sampling 2050:

- Biomimetic sampling systems using sustainable materials.
- Integration of biological materials into sampling systems – proteins, cells, intelligent antennae.
- Sampling, sensing and data processing integrated into single components.

- Ubiquitous sampling systems for biological fluids for medical diagnosis.

2) Sensors 2050:

- Most traditional sensors will be phased out and be replaced by miniature physical measurement systems, based on ion mobility, mass measurement or optical. This will be the size of current sensors.
- Nearly all sensors will be digitally integrated and where needed, linked to the cloud.
- For industrial processes, mains power will still dominate, but sensors will be developed with ultra-low power consumption that are able to scavenge energy from the environment.
- 2D and more exotic materials will become common place for consumer products, where price point and size are important. However, MOX materials will still be around and common.
- There will be a diversity in sensor manufacturing, with new companies dominating the consumer setting with the production of these next generation materials.
- Flexible and plastic based sensors will be developed– allowing sensors to be placed in any location.
- Wearable sensors will be more widely utilized, with more digital inclusion.
- Personal odor will be used as a means of identification.
- Wearable glasses that allow visualisation – the equivalent of thermal cameras for odors.
- Biological sensor systems will be developed, that contain replicated biological receptors
- Smell capture of environments to be replicated on display devices.

3) Data Analysis 2050:

- Electronic noses will include neuromorphic computing hardware and they will implement bioinspired computing. These computing solutions based on event driven signal processing and spike based neural networks will demand less computing power and they will be available in small form factor hardware for implementation in miniature sensing systems.
- Dramatic improvements in component tolerances will make calibration models available across identical instruments without or minimal additional calibrations
- eNoses size will decrease dramatically, and all the requirements regarding signal processing and machine learning will be integrated in single chip computers with minimum power requirements
- Databases related to the response of vertebrate's/insect's olfactory receptors (ORs) for a large palette of odorants will be widely available. The term olfactome will gain widespread use to represent the response of biological OR to a palette of odorants. International efforts will populate worldwide databases with the response of thousands of OR to millions of odorants.
- First demonstrators of realistic and bioinspired olfactory coding will appear. A proxy for the number of required channels will the number of active olfactory genes in humans. Odor analysis will allow to predict the response of human OR to this odor beforehand. This prediction will be enabled by machine learning models in large databases previously mentioned
- A side effect of the previous points and together with improvements in sensor technology, machine learning will allow to tailor the response of the sensor array to mimic the response of subsets of OR, to obtain a biomimetic enose output for particular biological species. Demonstrators for insect olfaction will appear.
- Brain computer interfaces for the olfactory system of dogs will allow to use the canine olfactory system for applications demanding maximum sensitivity and specificity. Dedicated signal processing for the acquired neural signals will allow interpretation and further processing of the dog olfactory response.

4) Applications 2050:

- eNose devices will be included in every household appliance, including the vacuum cleaner. Realtime odor identification and level will be reported to a home data center. Offending odors will be traceable to the generating source and event.
- eNose devices will be in every moving vehicle to accurately record the interior and exterior air quality. The exterior data will be routed to community processing nodes for second-to-second geo-mapping of problem conditions. Generators of the offending conditions will be identified, and appropriate action will then be possible to quickly mediate the problems.
- Every smartphone will include an eNose as an augmentation to the human nose. Specific options for the augmentation will be offered. For the elderly, general olfactory support will help them compensate for smell degradation. For those who work in special environments (like a chemical factory), a tailored eNose sensor array will be available.
- Robots, such as helpers or dogs, will have integrated eNoses and be able to recognise their owners based on smell.
- Small individual eNose units will be inexpensive and versatile for use in thousands of applications. They will use energy harvesting to operate for years without maintenance. They will be self-calibrating and capable of restoring their sensors to baseline operation millions of times before their end of life. All units will be disposable and biodegradable.

5) Standards 2050:

- The European and IEEE standards will be collaborating on joint projects.
- Fifty or more odor-monitoring and control standards will have been published with 30 or more them undergoing revision for continued relevance to the eNose manufacturers.
- Each year, 30 to 40 new Working Groups are formed, with 20 or more new standards being published.
- eNose manufacturers are finding the IEEE ICAP an important part of their efforts to develop great products with high market demand. Twenty or more ICAP sites will be in operation.

- More than a dozen eNose companies will be publicly traded and viewed as very successful commercial entities and green employers.

C. Forecast for 2075

1) Sampling 2075:

- Autonomous systems for sampling, sensing and remedial action.
- Bioengineered sampling and sensing systems.
- Intelligent materials for sampling and sensing capable of self-healing and self-reproduction.
- Fully biomimetic systems.

2) Sensors 2075:

- eNoses will be integrated into the fabric of structures and be invisible. It will become a national standard to have smart odor analysis systems built into our environment.
- Ability to control materials and material properties at the atomic level will allow many materials to operate as chemical sensors – measuring physical and chemical properties of molecules.
- We will move around the environment with our clothes, buildings and personal transport devices. These systems will be able to track individuals based on their odor profile.
- Global odor maps will be created and updated on a second-by-second basis.
- Bio-replacement human noses for those who lose their sense of smell.
- Bio-enhancement of the human nose to be the same as a dog or beyond.

3) Data Analysis 2075:

- The problem of olfactory coding will be fully understood. Deep learning will be available to predict in-silico odor perception for arbitrary odorant molecules not yet synthesized.
- Electronic noses will implement theoretical neuroscience models of the olfactory circuitry in neuromorphic hardware. This implementation will take as inputs the predicted OR response from sensor signals (either by direct OR biosensors or biomimetic inorganic sensors plus signal processing), but beyond that it will be capable to predict non-linear and dynamic synergistic and antagonistic response for a full biomimetic prediction of human perception experience.
- First attempts to use chemical sensors for olfactory prothesis for anosmic people. While successful, the olfactory experience will be limited in richness and span. However, people will have augmented olfaction capabilities being able to perceive dangerous gases such as CO.

4) Applications 2075:

- eNose units of varying complexities are ubiquitous in our indoor and outdoor environment. As old eNose devices fail and fade away, new units are routinely introduced into the same or a nearby location. Cloud-based data centers collect vast amounts of data. Each eNose includes its health status in every report. Replacement geo-maps will show users where new units are needed, and what capabilities should be included in replacement units.
- Since each eNose will be biodegradable, collection centers will only be needed if recycling any of the eNose materials is profitable or helps maintain the health of the planet in any manner.

5) Standards 2075:

- Hundreds of odor-monitoring and control standards will be available to support eNose performance claims by the manufacturers. Purchasers of eNose devices that are certified to meet the published standards are happy customers and will return decade after decade to purchase newer, more sophisticated units.
- More than 50 ICAP testing sites are available, reaching all continents.
- There are more than 100 successful eNose companies, with mergers and acquisitions occurring multiple times each year.

V. Conclusion

The artificial olfaction field has been built on a strong foundation. The founders of the field have set us on a creative voyage that is well underway. Past efforts have proven that eNose technology can solve odor monitoring and control problems in a wide variety of applications. Commercial success has been challenging because specific designs that are cost-limited, trustworthy, and tailored to unique markets are not easy to find. As sensors become more accurate and interfacing electronics get smaller, while both get cheaper and more reliable, those hard-to-find applications will emerge, and the commercial firms will flourish. We hope that the inquisitive young minds of current and future brilliant science and engineering students will see artificial olfaction as a green technology that can help save the world.

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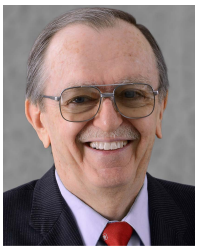

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