

HIGHLY SELECTIVE NO₂ GAS SENSOR OPERATING AT ROOM TEMPERATURE WITH PPB-LEVEL DETECTION SENSITIVITY BASED ON ZnO NANOPARTICLES

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Abstract

In this work, authors reported a high-selectivity nitrogen dioxide (NO₂) gas sensor that operates directly at room temperature without catalyst or illumination. The NO₂ gas sensor was based on ZnO nanoparticles, which were synthesized using a simple solution method, ensuring scalability in mass production. Crucially, the sensor exhibited high sensitivity and selectivity towards NO₂, achieving parts-per-billion (ppb) level detection limits. At 23°C, the sensor showed a sensitivity of 4.2% with the response time and recovery time are 120 s and 185 s, respectively, under 5 ppb NO₂, while the interaction with other gases such as H₂S, NH₃, SO₂, CH₄, C₃H₈, and CO₂ was negligible. This room-temperature, easily fabricated sensor offered a promising solution for cost-effective, real-time NO₂ monitoring in various applications, particularly those requiring high sensitivity at low NO₂ concentrations.

Keywords: NO₂; ZnO nanoparticles; room-temperature; ppb level.

1. Introduction

Nitrogen dioxide (NO₂) is a pervasive air pollutant with significant implications for human health and the environment. Exposure to elevated NO₂ levels is linked to respiratory illnesses, cardiovascular diseases, and increased mortality rates, particularly among vulnerable populations such as children and the elderly [1]-[3]. Furthermore, NO₂ plays a critical role in the formation of acid rain, smog, and other secondary pollutants that degrade air quality and damage ecosystems [4]-[6]. Given its detrimental impact, accurate, reliable, and cost-effective monitoring of NO₂ levels is paramount for effective pollution control strategies and public health protection.

Current NO₂ sensing technologies, while offering varying degrees of sensitivity and selectivity, often suffer from significant limitations that restrict their widespread implementation. Many established methods require operating conditions far from ambient, introducing substantial challenges for practical applications.

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Many research have focused on semiconductor-based NO₂ gas sensors, predominantly utilizing metal oxide nanomaterials such as tin oxide (SnO₂), tungsten oxide (WO₃), and zinc oxide (ZnO) [7]-[12]. These materials exhibit changes in their electrical conductivity upon exposure to NO₂, a phenomenon exploited in chemiresistive sensing. However, a common limitation of these chemiresistive sensors is their relatively low sensitivity at room temperature. The interaction between NO₂ molecules and the semiconductor surface often requires thermal activation to overcome energy barriers and enhance the adsorption process, leading to a noticeable improvement in sensitivity at high temperatures [13], [14]. This necessity for heating elements significantly increases the power consumption, size, and cost of the sensor, thus limiting their suitability for portable and low-power applications.

Furthermore, many high-sensitivity NO₂ sensors rely on the use of catalysts to improve their response characteristics. Noble metals like platinum (Pt) and palladium (Pd) are frequently employed as catalysts, facilitating the oxidation of NO₂ and enhancing the signal generated by the sensor [15]-[17]. While effective, the inclusion of these precious metals significantly increases the cost of the sensor, making large-scale deployment prohibitively expensive. Moreover, the long-term stability and durability of catalyst-based sensors can be a concern, as the catalyst can be poisoned or deactivated over time due to exposure to contaminants in the environment or due to the reaction with NO₂ itself [18], [19].

Another established strategy for enhancing the sensitivity of NO₂ sensors involves photocatalytic oxidation using ultraviolet (UV) illumination. UV light provides the energy required to excite the semiconductor material, thereby promoting the adsorption and oxidation of NO₂ molecules on the sensor's surface [20]-[22]. The resulting changes in electrical conductivity or optical properties are then measured to determine the NO₂ concentration. However, this approach introduces additional complexity and cost associated with integrating a UV light source and power supply into the sensor system. The reliance on UV light also restricts the operational flexibility of the sensor, making it less adaptable to diverse environmental conditions. Furthermore, the lifetime of the UV source itself poses a practical limitation.

This study introduces a highly selective nitrogen dioxide gas sensor that operates at room temperature without the need for catalysts or illumination with ppb-level detection. The sensor is based on ZnO nanoparticles, which are synthesized using a solution method that allows for scalable mass production. This easily fabricated, room-temperature sensor provides a promising approach for cost-effective, real-time monitoring of NO₂, especially in applications that demand high sensitivity at low concentrations.

2. Experiment

2.1. Synthesis of ZnO nanoparticles

The ZnO nanoparticles were synthesized through a simple precipitation method (Fig. 1). Initially, 30 ml $\text{Zn}(\text{NO}_3)_2$ 0.1M was added gradually to the 20 ml NaOH 1 M. The mixture was stirred for 2h then the precipitate was collected by centrifugation, washed several times with deionized water, and dried at 80°C for 24h. The product was then annealed at 400°C for 2h.

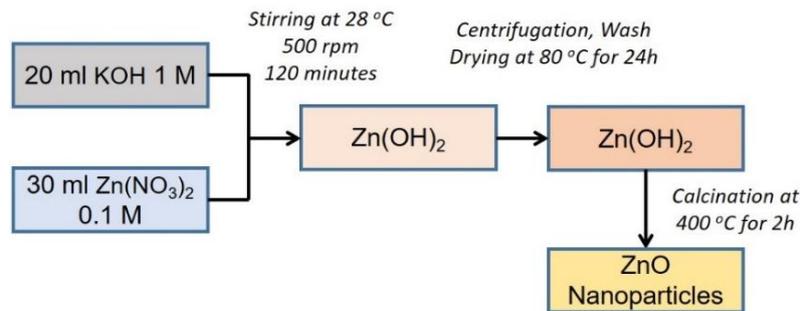


Fig. 1. Schematic illustration of the synthesis procedure for ZnO nanoparticles.

2.2. Sensor fabrication and gas sensing measurements

The ZnO NP-based device was prepared using a spin coating technique and deposited on a silicon substrate with a Pt electrode (Fig. 2). Initially, a small amount of as-synthesized ZnO powder was dispersed in 1 ml of deionized water. The mixture was sonicated multiple times to achieve uniformity. Subsequently, the homogeneous solution was dropped onto a Pt/ SiO_2 /Si substrate ($0.5 \times 0.5 \text{ cm}^2$) and spin-coated at 200 rpm for 20 seconds, followed by calcination at 400°C for 120 minutes.

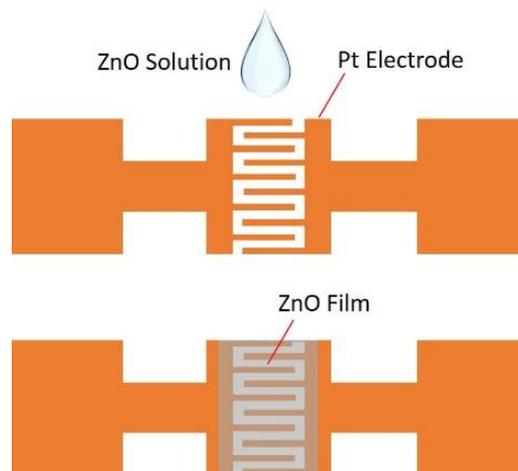


Fig. 2. Schematic diagram of the ZnO NPs sensor structure.

The ZnO device's gas-sensing characteristics were evaluated through dynamic gas testing in a light-shielded chamber. Dry air served as the carrier gas, flowing directly onto the ZnO device's surface at a total rate of 300 sccm. Gas sensing tests were carried out at room temperature and in dry conditions (< 3% RH). Gas-sensing measurements were conducted using a Keithley 4200SCS. The response value (S) is calculated as

$$S = 100\% \times \frac{|R_a - R_g|}{R_a}$$

where R_a and R_g represent the device's resistance in air and target gas, respectively.

Response and recovery times are defined as the duration required for 90% of the total resistance change during the response and recovery phases.

2.3. Characterization

The as-synthesized sample's morphologies were analyzed using field emission electron microscopy (FE-SEM - JSM-6701F) at a 10 kV accelerating voltage. The sample's phase composition, crystallinity, and chemical composition were evaluated by X-ray diffraction (XRD) using CuK α radiation ($\lambda = 1.54178 \text{ \AA}$) at potential and current of 40 kV and 30 mA, respectively.

3. Result and discussion

3.1. Morphology and structure

Figure 3 presents the XRD pattern of the as-synthesized ZnO nanoparticles. All these diffraction peaks verified that the as-synthesized product is standard hexagonal crystal phase of ZnO (JCPDS No. 01-070-2551). The major peaks with reflections observed at 2θ of 31.78° , 34.43° , and 36.30° are consistent with the corresponding to the (100), (002), and (101) crystal planes of ZnO wurzite structure.

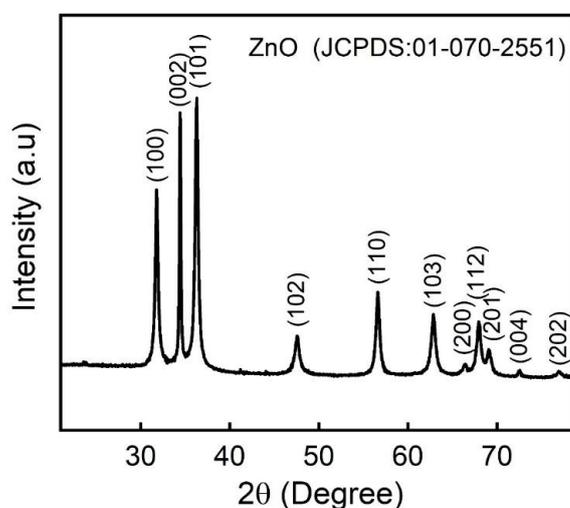


Fig. 3. Powder XRD results of ZnO nanoparticles.

Figures 4 (a, b) show the uniform distribution of the ZnO nanoparticles with diameters ranging from 90 nm to 110 nm.

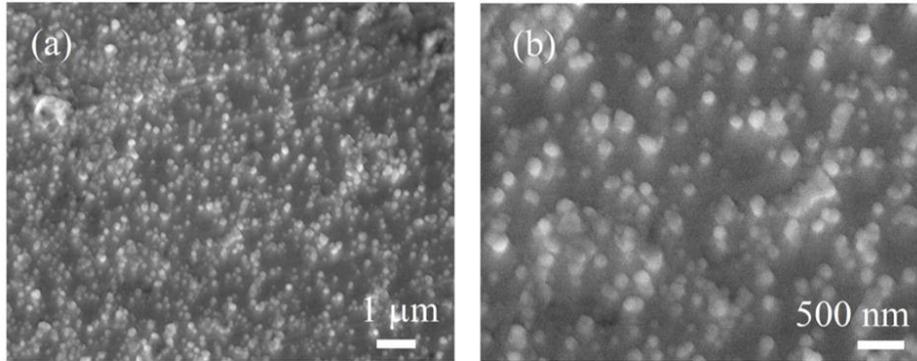


Fig. 4. FE-SEM images of the synthesis ZnO nanoparticles: (a) low magnification; (b) high magnification.

3.2. Gas sensing properties

The gas test was performed at room temperature with various target gases, including NO₂, H₂S, NH₃, CH₄, C₃H₈, CO₂, and SO₂, to evaluate the selectivity of the ZnO NPs device. The dynamic response of the ZnO NPs device to these gases at room temperature is illustrated in Fig. 5.

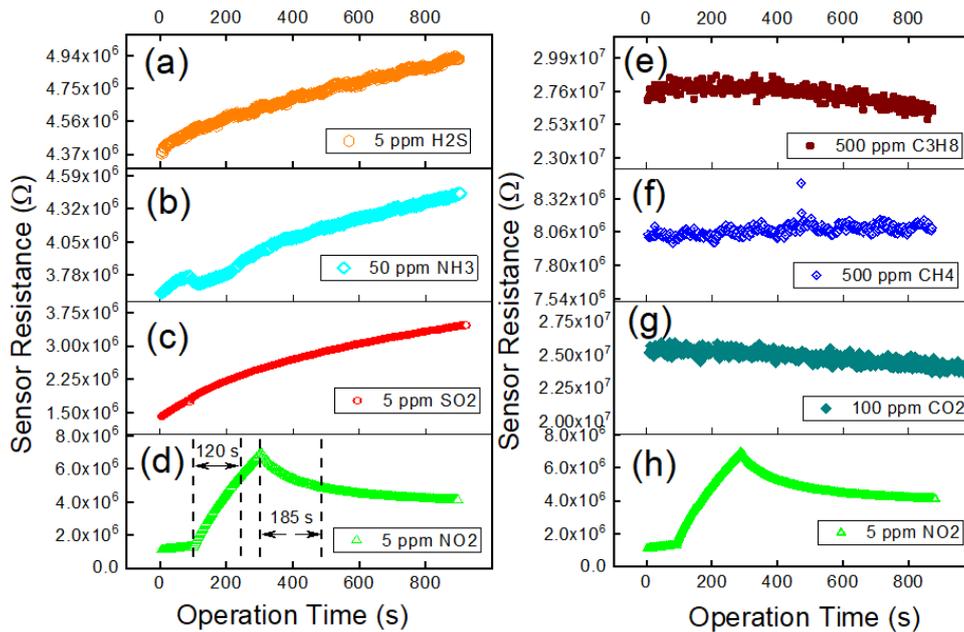


Fig. 5. Transient response curves of the device based on the ZnO nanoparticles to different gases at room temperature: (a) 5 ppm H₂S; (b) 50 ppm NH₃; (c) 5 ppm SO₂; (d) 5 ppm NO₂; (e) 500 ppm C₃H₈; (f) 500 ppm CH₄; (g) 10 ppm CO₂; (h) 5 ppm NO₂.

The results showed that the sensor exhibited sensitivity only to NO_2 gas under identical testing conditions. This selectivity may be attributed to the varying reaction energies of the test gases on the ZnO NPs surface [23], [24]. The device's resistance increased upon exposure to NO_2 , indicating n-type semiconducting behavior in response to an oxidizing gas (Fig. 5h). The response and recovery times of the device toward 5 ppm NO_2 were determined to be 120 s and 185 s, respectively (Fig. 5d).

The dynamic response of the ZnO nanoparticle-based sensor to NO_2 concentrations ranging from 25 ppb to 1000 ppb at ambient temperature was presented in Fig. 6a. Upon removal of NO_2 , the sensor exhibited rapid and reversible response with full baseline recovery. Calibration curves derived from the dynamic response data were shown in Fig. 3b, revealing an increase in sensor response with escalating NO_2 concentrations. The relationship between sensor response and NO_2 concentration is linear, with a correlation coefficient (R^2) of 0.995.

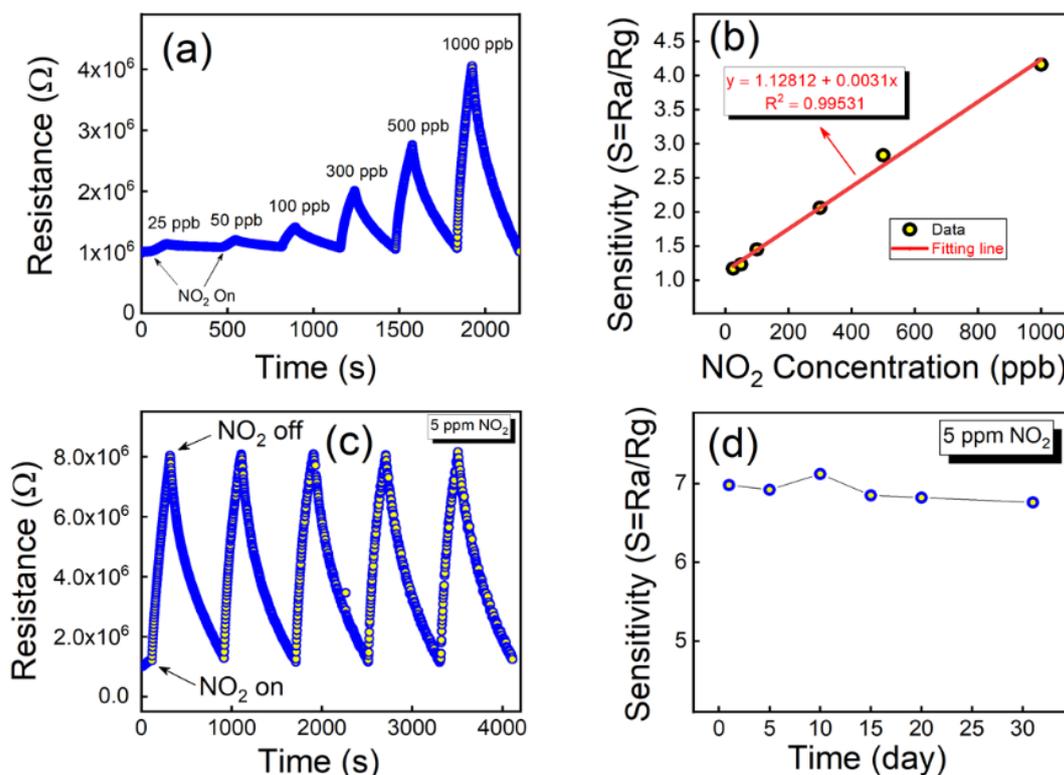


Fig. 6. Response of the ZnO nanoparticles device upon the exposure to NO_2 gas at room temperature: (a) Dynamic response of the device to various concentrations of NO_2 ; (b) relationship between the response of the device and NO_2 concentration; (c) repeatability of the device toward 5 ppm NO_2 ; (d) long-term stability of the device toward 5 ppm NO_2 .

Short-term and long-term stability are critical parameters for gas sensors. Fig. 6c illustrated the response and recovery profile for 5 ppm NO₂ over five consecutive on/off cycles, confirming excellent repeatability and short-term stability. Long-term stability was further validated through six tests conducted over 30 days, with continuous measurements of the sensor response to 5 ppm NO₂ maintaining a consistent response level with approximately 4.2% variation (Fig. 6d).

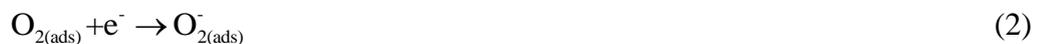
The comparison of various NO₂ room-temperature gas sensors is shown in Tab. 1 [23], [25]-[29]. Compared to conventional sensors operating at room temperature, our ZnO nanoparticles sensor demonstrated rapid response and recovery durations for NO₂.

Tab. 1. Comparison of various NO₂ room-temperature gas sensors [25]-[29]

Material	Operating temperature	Concentration (ppm)	T _{Res} /T _{Rec} (s)	Response (%)	Detection limit (ppm)	Ref.
BiI ₃ nanoplate	Violet light at RT	1	32/71	7.96	0.25	[23]
SnS ₂ /rGO	RT	1	240/400	75.0	0.2	[25]
Cs ₂ SnI ₆ thin film	RT (25°C)	20	247/818	35.0	3.0	[26]
GaSe _{0.58} O _{0.42}	RT	6	48/378	87.1	0.05	[27]
ZnO@M-TiO ₂	RT	1	128/175	250.0	0.5	[28]
Au-ZnO NWs	UV irradiation at RT (25°C)	1	160/370	130.0	0.02	[29]
ZnO nanoparticle	RT (23°C)	1	120/185	320.0	0.25	This work

3.3. Gas sensing mechanism

The proposed NO₂ gas-sensing mechanism of the ZnO nanoparticle-based sensor was illustrated in Fig. 7. At ambient temperature, oxygen molecules adsorb onto the ZnO NP surface, capturing electrons and forming stable chemisorbed oxygen ions O_{2(ads)}⁻, as depicted in Fig. 7a. The adsorption process at room temperature can be described by the following reactions [23], [30]:



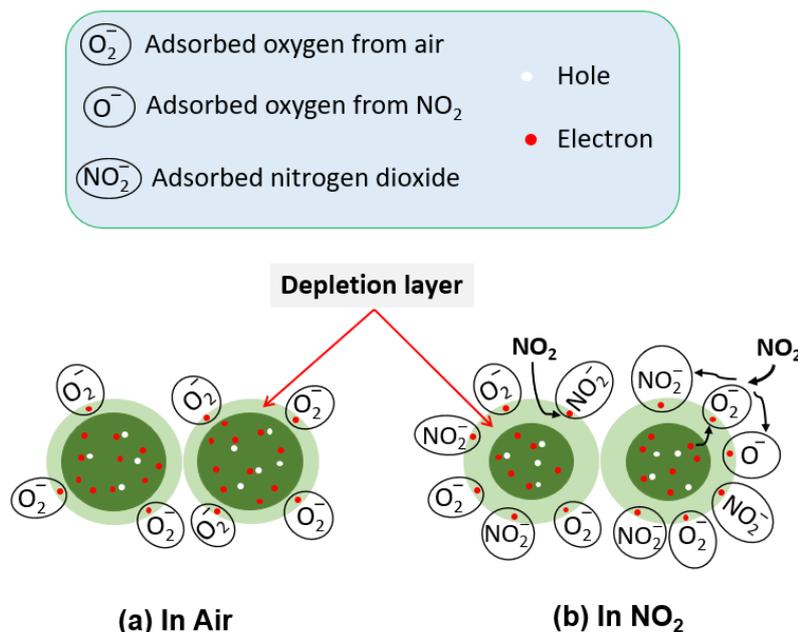
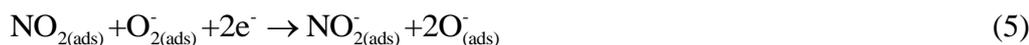


Fig. 7. Schematic of proposed NO₂ sensing mechanism of ZnO NPs based sensor at room temperature: (a) in air; (b) in NO₂.

Upon exposure to NO₂, NO₂ molecules adsorb onto the ZnO NP surface and extract electrons from the conduction band, forming NO_{2(ads)}⁻ ions (Eq. (5)). Additionally, due to the higher electronegativity of NO₂ compared to oxygen, NO₂ molecules may react with pre-adsorbed O_{2(ads)}⁻ ions on the ZnO surface, further capturing conduction band electrons to form NO_{2(ads)}⁻ and O_(ads)⁻ species (Eq. (6)). This process expands the electron depletion layer (Fig. 7b), increasing the material's resistance due to a reduced carrier concentration (Fig. 6c). The relevant reaction processes are expressed as follows [30], [31]:



4. Conclusion

In conclusion, ZnO nanoparticles (NPs) with an approximate diameter of 100 nm were successfully synthesized using a simple precipitation method. The sensor fabricated from these ZnO NPs exhibited effective NO₂ detection at room temperature, characterized by high selectivity, excellent reproducibility, and a low detection limit at

the ppb level, without requiring noble metal doping or light irradiation. These findings position ZnO NP-based sensors as promising candidates for future gas-sensing applications. Further research is warranted to investigate the NO₂ sensing performance of ZnO NP-based sensors under varying humidity conditions to enhance their suitability for practical room-temperature NO₂ detection.

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CẢM BIẾN KHÍ NO₂ CÓ ĐỘ CHỌN LỌC CAO HOẠT ĐỘNG Ở NHIỆT ĐỘ PHÒNG VỚI KHẢ NĂNG NHẠY KHÍ Ở MỨC PPB DỰA TRÊN CÁC HẠT NANO ZnO

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Tóm tắt: Trong nghiên cứu này, các tác giả báo cáo về một cảm biến khí nitrogen dioxide (NO₂) có độ chọn lọc cao, hoạt động trực tiếp ở nhiệt độ phòng mà không cần chất xúc tác hoặc chiếu sáng. Cảm biến NO₂ được chế tạo dựa trên các hạt nano ZnO, được tổng hợp bằng phương pháp dung dịch đơn giản, đảm bảo khả năng sản xuất ở quy mô lớn. Đặc biệt, cảm biến thể hiện độ nhạy và độ chọn lọc cao đối với NO₂, đạt giới hạn phát hiện ở mức cỡ phần tỉ (ppb). Ở nhiệt độ 23°C, cảm biến cho độ nhạy 4,2% với thời gian đáp ứng và thời gian phục hồi lần lượt là 120 giây và 185 giây đối với 5 ppb NO₂, trong khi khả năng tương tác với các loại khí khác như H₂S, NH₃, SO₂, CH₄, C₃H₈ và CO₂ là không đáng kể. Với quy trình chế tạo đơn giản và khả năng hoạt động ở nhiệt độ phòng, cảm biến này là một giải pháp tiềm năng cho việc giám sát NO₂ theo thời gian thực, đặc biệt là các ứng dụng yêu cầu độ nhạy cao ở nồng độ NO₂ thấp.

Từ khóa: NO₂; hạt ZnO; nhiệt độ phòng; nồng độ cỡ phần tỉ (ppb).

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