

MEASUREMENTS OF EFFECTIVE DIFFUSION COEFFICIENTS OF HELIUM AND HYDROGEN THROUGH GYPSUM*

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ABSTRACT

An experimental apparatus, which was based on the ¼-scale garage previously used for studying helium release and dispersion in our laboratory, was used to obtain effective diffusion coefficients of helium and hydrogen (released as forming gas for safety reasons) through gypsum panel. Two types of gypsum panel were used in the experiments. Helium or forming gas was released into the enclosure from a Fischer burnerⁱⁱ located near the enclosure floor for a fixed duration and then terminated. Eight thermal-conductivity sensors mounted at different vertical locations above the enclosure floor were used to monitor the temporal and spatial gas concentrations. An electric fan was used inside the enclosure to mix the released gas to ensure a spatially uniform gas concentration to minimize stratification. The temporal variations of the pressure difference between the enclosure interior and the ambience were also measured. An analytical model was developed to extract the effective diffusion coefficients from the experimental data.

KEYWORDS: Diffusion coefficient; Gypsum; Helium; Hydrogen

INTRODUCTION

In a future hydrogen economy, it is likely that hydrogen-powered fuel-cell vehicles will be parked inside residential garages. Therefore, it is imperative to understand how accidentally released hydrogen from these vehicles is dispersed inside a garage and the degree of potential hazard as a result of the release. To study the hydrogen dispersion and mixing processes and to develop hazard mitigation strategies, a ¼-scale two-car garage made of poly(methyl methacrylate) (PMMA) was used to conduct reduced-scale experiments in the laboratory [1,2]. In addition, for operational and laboratory safety reasons, helium was used as a surrogate for hydrogen. This approach is well documented in the literature [e.g., 3-6].

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In the United States, the interior walls and ceilings of most residential garages are covered with gypsum panels. Since gypsum panel is a highly porous material, volatile organic compounds (VOCs), moisture, and other low molecular weight vapors typically found in a garage can diffuse through gypsum [7-9]. Hydrogen, which has the lowest atomic weight in the periodic table, is expected to diffuse readily through gypsum. The diffusion of hydrogen through gypsum panel may be important because of the large gypsum panel surface area used in garages and could facilitate the penetration of hydrogen into the adjacent living areas of the house from the garage and build up of a concentration sufficient to represent an unwanted hazard. Conversely, the gypsum panel could also allow hydrogen to diffuse outside and help to alleviate concentration build-up in the garage. In order to estimate the mass diffusion rate of hydrogen through gypsum panel, an effective diffusivity of hydrogen through gypsum panel is needed. Although the diffusion coefficients of VOCs and moisture through gypsum have been extensively studied in the literature [7-9], no data on hydrogen and helium diffusion through gypsum panel was identified.

This paper describes an experimental technique to measure an effective diffusion coefficient of helium or hydrogen through gypsum panel, which made use of the ¼-scale two-car garage that was previously used to study helium mixing and dispersion [1,2]. The technique is based on the following analysis and is analogous to the dual chamber (twin chamber) method used to study VOC and moisture diffusion through building materials [8-9] and gaseous diffusion through a polymer film [10]. In our method, one chamber was the PMMA enclosure, and the ambience was considered to be the other chamber.

EXPERIMENTS

Fig. 1 is a photograph of the experimental set-up, which was derived from the ¼-scale two-car garage that was used to study mixing and dispersion of helium [1,2]. The reduced-scale garage with inside dimensions of 0.75 m (height) × 1.5 m (width) × 1.5 m (length) was constructed of PMMA sheets (1.27 cm thick) with the front wall replaced by a section of gypsum panel (1.58 m × 0.82 m) for the measurement of effective diffusion coefficient of helium and hydrogen through gypsum. The PMMA sheets were glued together. A 2.54 cm wide PMMA flange was attached around the outside edge of the open end of the PMMA enclosure. The gypsum panel was attached to the PMMA enclosure with a supporting metal frame and rubber gasket using C-clamps and stopcock grease to form a tight seal. The backside (the side with liner paper) of the gypsum panel was oriented toward the enclosure interior, and the front side with natural-finish face paper was exposed to the ambience.

A Fischer burner with a 3.6 cm opening was used as a convenient way to release helium or hydrogen in the form of forming gasⁱⁱⁱ (0.0413 volume fraction of hydrogen in nitrogen) into the enclosure. The burner, the outlet of which was 20.7 cm above the enclosure floor, was placed on the floor at the center of the enclosure. A 1.9 cm threaded hole was cut out of a small PMMA cover plate mounted onto the

ⁱⁱⁱ For safety reasons, forming gas was used in lieu of pure hydrogen.

gypsum panel to prevent pressure build-up during the helium or hydrogen release into the enclosure. A small electric fan was installed inside the enclosure to continuously mix the released helium or forming gas to ensure a uniform helium or hydrogen concentration and to prevent stratification in the enclosure. Two types of gypsum panels, USG SHEETROCK® Brand 1.3 cm (1/2") thick and USG SHEETROCK® Brand FIRECODE® Core Type X 1.6 cm (5/8") thick, were used. Experiments were conducted by injecting helium or hydrogen at a fixed rate using a mass flow controller for an extended period of time (≥ 1 h). In some cases, the helium or hydrogen concentration attained a steady state. The helium or hydrogen injection was then terminated, and the enclosure hole was capped. Eight thermal-conductivity sensors (Xensor Integration), with seven positioned at fixed vertical locations above the floor (9.3 cm, 18.5 cm, 27.6 cm, 37.2 cm, 46.6 cm, 55.9, and 65.0 cm with Sensor #1 being the lowest and Sensor #7 the highest from the floor) and one (Sensor #8) freely placed at variable locations during different tests, were installed inside the enclosure to monitor the uniformity of helium or hydrogen concentrations at different heights. A differential pressure transducer (1333 Pa MKS Baratron electronic manometer) placed in the front wall at mid height was used to measure the pressure difference between the enclosure interior and the ambience. Sensor and pressure transducer outputs were logged using a personal computer and a data acquisition system. Detailed descriptions of sensor locations, calibration, operation, and measurement uncertainty (relative uncertainty of 1 %) can be found in Pitts et al. [1,2]

ANALYSIS

Fig. 2 shows a schematic of the model used for the analysis. The major assumptions used in the analysis are:

- The helium or hydrogen concentration is uniform in the enclosure.
- A quasi steady-state linear concentration profile is established within the gypsum. This approximation is justified even though the concentration in the enclosure is changing with time because the enclosure volume is much greater than that of gypsum [10].
- Diffusion of helium or hydrogen through PMMA is assumed to be negligible.
- Although the edges of the gypsum panel are not covered with paper, diffusion through the exposed edges is assumed to be negligible because the total edge area constitutes a very small fraction (about 5 %) of the total mass transfer area.
- Helium and hydrogen are treated as ideal gases.

The helium or hydrogen molar balance on the enclosure is:

$$\frac{dN}{dt} = \dot{n}_f - \dot{n}_c - \dot{n}_d''A \quad (1)$$

where N is the amount of helium or hydrogen (mol) inside the enclosure, \dot{n}_f is the helium or hydrogen molar flow rate into the enclosure (mol/s), \dot{n}_c is the convective helium or hydrogen molar flow rate out of the enclosure through the small enclosure opening (mol/s), \dot{n}_d'' is the unidirectional diffusive molar flux through the gypsum panel (mol/s/m²), t is the time (s), and A is the gypsum panel surface area for diffusion (m²).

The quasi-steady diffusive molar flux can be approximated and expressed by:

$$\dot{n}_d'' = D_e \frac{(C - C_\infty)}{\delta} \quad (2)$$

where D_e is an effective helium or hydrogen diffusion coefficient through the gypsum panel (m²/s), C is the helium or hydrogen molar concentration in the enclosure (mol/m³), C_∞ is the helium or hydrogen molar concentration in the surrounding (mol/m³), and δ is the thickness of the gypsum panel (m). In writing Eq. (2), one could view D_e/δ as an overall mass transfer coefficient with negligible gas-phase convective mass transfer on both sides of the gypsum panel. If the enclosure interior and the ambience are assumed to be completely mixed, the gas-phase convective mass transfer to and from the gypsum panel surfaces can be considered less important than mass diffusion within the gypsum panel [9].

Eq. (1) can be written in terms of the amount-of substance (mole) fraction of helium or hydrogen as follows.

$$\frac{d}{dt} \left(\frac{yPV}{RT} \right) = (\dot{n}_f - \dot{n}_c) - D_e A \frac{(yP - y_\infty P_\infty)}{\delta RT} \quad (3)$$

$$\frac{PV}{RT} \frac{dy}{dt} + \frac{V}{RT} y_a \frac{dP}{dt} = (\dot{n}_f - \dot{n}_c) - D_e A \frac{(yP - y_\infty P_\infty)}{\delta RT} \quad (4)$$

where y is the mole fraction of helium or hydrogen in the enclosure, y_∞ is the helium or hydrogen mole fraction in the surrounding, V is the enclosure volume (m³), R is the universal gas constant (= 8.314 J/mol/k), P is the enclosure pressure (Pa), P_∞ is the ambient pressure (Pa), and T is the enclosure temperature (K).

With the small hole on the gypsum panel open during helium or hydrogen injection to equilibrate the enclosure pressure with the ambient pressure, P does not change significantly and can be approximated by $P \approx P_\infty \approx \text{constant}$ and with $y_\infty \approx 0$ and $\dot{n}_c \ll \dot{n}_f^{\text{iv}}$, Eq. (4) can be approximated by:

$$\frac{dy}{dt} = \left(\frac{\dot{n}_f RT}{PV} \right) - \frac{D_e A y}{V \delta} \quad (5)$$

Solving Eq. (5) with the initial condition of $y = 0$ at $t = 0$, we obtain

$$y = \frac{\dot{n}_f RT \delta}{P D_e A} \left(1 - e^{-\frac{D_e A}{V \delta} t} \right) \quad (6)$$

When a steady state is reached ($t \rightarrow \infty, y = y_{ss}$), Eq. (6) yields

$$D_e = \frac{\dot{n}_f RT \delta}{P A y_{ss}} \quad (7)$$

For a given helium or hydrogen injection rate \dot{n}_f and by measuring y as a function of t , Eq. (6) can be used to obtain the effective helium or hydrogen diffusion coefficient in gypsum panel by a best-fit of the $y(t)$ data in the form of $a(1 - e^{-bt})$, where a and b are optimized curve-fit parameters. Alternatively, Eq. (7) can be used to estimate D_e by simply measuring the steady-state helium or hydrogen mole fraction y_{ss} in the enclosure when a steady state is attained within the experimental run time.

When the helium or hydrogen injection was terminated, and the small opening was capped, helium or hydrogen continued to diffuse through the gypsum panel and out of the enclosure, while air diffused back through the gypsum panel and into the enclosure. Since helium or hydrogen diffusion was faster than the diffusion of air, the pressure inside the enclosure initially decreased sharply below ambient pressure (see Figs. 4 and 6 below). As more and more air diffused through the gypsum panel into the enclosure, the helium or hydrogen concentration gradient for diffusion decreased due to the dilution with the diffused air and the decreasing amount of helium or hydrogen in the enclosure. Eventually, the enclosure pressure ceased to decrease when the diffusion of air was balanced by the diffusion of helium or hydrogen. As air continued diffusing into the enclosure and helium diffusing out, the enclosure pressure slowly recovered and increased to equilibrate with the ambient pressure. This phenomenon was similar to that observed in the pioneering work of Thomas Graham who used a diffusion tube to study the diffusion of gases in 1829 [10].

^{iv} $\dot{n}_c = y C_d \rho_m A_c \sqrt{2(P - P_\infty)/\rho}$, where C_d is the discharge coefficient, ρ_m is the molar density of the mixture in the enclosure, ρ is the mixture mass density, and A_c is the area of the small hole. Since $C_d \sim O(1)$, $\rho \sim O(1)$, $\rho_m \sim O(10)$, $A_c \sim O(10^{-4})$, $y \sim O(10^{-1})$ for helium, and $P - P_\infty \sim O(1)$, $\dot{n}_c \sim O(10^{-4})$. For hydrogen, $y \sim O(10^{-2})$, $\dot{n}_c \sim O(10^{-5})$.

RESULTS AND DISCUSSION

Typical experimental results are illustrated in Figs. 3 and 4, which show the temporal variations of helium concentrations in the enclosure measured by the eight sensors and the corresponding temporal variations of pressure differences, $P - P_{\infty}$, between the enclosure interior and the ambience with a helium injection rate of $\dot{n}_f = 2.43 \times 10^{-3}$ mol/s. The uniformity of helium concentrations (little stratification) is reflected by the eight sensor readings in the figures.

There are two stages in Fig. 3. The first stage signifies the initial increase in helium concentration and the subsequent attainment (the plateau region) of a steady-state helium concentration during the injection of helium into the enclosure. Note that the attainment of a steady state was not necessary for the experiments in order to extract the effective diffusion coefficients. The second stage, during which the helium concentration in the enclosure decays, results from the termination of helium injection and the closure of the small hole on the gypsum panel. There was a very small pressure difference recorded during the first stage^{iv}, which implies there was little flow through the small enclosure opening, which justifies the assumption of $\dot{n}_c \approx 0$ used in deriving Eq. (4). The small opening on the gypsum panel simply acted to equalize the interior and exterior pressure to prevent pressure build-up inside the enclosure during helium injection. Once the helium injection was terminated, and the enclosure hole was capped, the enclosure pressure first decreased rapidly, reached a minimum, and recovered to ambient pressure, as discussed in the previous section. Figs. 5 and 6 shows respectively the concentration and pressure results for $\dot{n}_f = 1.02 \times 10^{-2}$ mol/s. A higher helium release rate results in a higher concentration gradient across the gypsum panel and a faster diffusion rate, thus causing the pressure difference to reach a more negative value (cf. Figs. 4 and Fig.6).

Figs. 7 and 8 show the experimental results of one test in which the backside (facing towards the enclosure interior) of the gypsum panel was coated with a layer of primer and two layers of latex semi-gloss white wall paint. In this test, the helium injection was terminated before the attainment of a steady-state. It can be seen from Fig. 7 that the helium concentrations are higher than those obtained using unpainted gypsum panels (see Fig. 5) with the same helium injection rate. In addition, the enclosure pressure recovers much more slowly after the termination of helium flow when compared to the results from unpainted gypsum panels (Fig. 8 vs. Fig. 6).

Since the helium concentration is uniform and there is insignificant helium stratification in the enclosure due to effective mixing using the electric fan, the average values of the concentrations from the eight sensors were used to obtain the effective diffusion coefficients using Eq. (6) and the best-fit parameters a and b using SigmaPlot[®] (version 10), a commercial graphing and data analysis software. An example of the curve-fit results using the data from Fig. 3 is shown in Fig. 9. Note that only the concentration data during the helium injection is needed to extract the effective diffusion coefficient.

With $T = 294$ K, $A = 1.09$ m², $V = 1.69$ m³, and $P = 0.101 \times 10^6$ Pa, the calculated effective diffusion coefficients of helium through gypsum panel are summarized in Table 1 for a number of experiments. The third and fourth columns of the table list the values of D_e ^v obtained from the best-fit parameters a and b respectively (see Eq. (6)); the averages of these two D_e values are tabulated in the last column of the table. For the low helium injection rate (2.43×10^{-3} mol/s), the derived D_e values are lower than those obtained using the high injection rate (1.02×10^{-2} mol/s). This could be due to the fact that the assumption of $\dot{n}_c \ll \dot{n}_f$ used to derive Eq. (5) may not be appropriate for the low injection rate, which is an order of magnitude larger than \dot{n}_c ; $\dot{n}_c \sim O(10^{-4})$ vs. $\dot{n}_f \sim O(10^{-3})$.

For the high helium injection rate, the estimated effective diffusion coefficient has a mean value of 1.4×10^{-5} m²/s ($n = 4$, where n is the number of measurements) with a relative standard deviation of 1.4 % for regular gypsum panel and a mean value of 1.3×10^{-5} m²/s ($n = 8$) with a relative standard deviation of 0.6 % for Type X gypsum panel. There is statistically significant difference between the mean values of the effective helium diffusion coefficients through regular and Type X gypsum panels ($P^{vi} \ll 0.001$ using two-sample t -test [11]). The diffusion coefficient through painted gypsum panel is about a factor of four smaller than that of the virgin gypsum panel.

Figs. 10 and 11 show the results of an experiment using forming gas and Type X gypsum panel. In this test, the forming gas injection was terminated before the attainment of a steady-state hydrogen concentration. Table 2 summarizes the effective diffusion coefficients through gypsum obtained for hydrogen. An average effective hydrogen diffusion coefficient of 1.4×10^{-5} m²/s ($n = 2$) was obtained with a relative standard deviation of 5.4 %.

Table 3 compares the effective diffusion coefficients of three VOC materials (toluene, formaldehyde, and decane) and water vapor through calcium silicate (a major component of gypsum) and gypsum reported in the literature with the effective diffusion coefficients of helium and hydrogen obtained in this study. The effective diffusivities of helium and hydrogen through gypsum panel are an order of magnitude larger. For reference, the diffusivity of helium in air at 3 °C is 6.2×10^{-5} m²/s [10], and that of hydrogen in air at 0 °C is 6.1×10^{-5} m²/s [10].

CONCLUSIONS

Effective diffusion coefficients of helium and hydrogen (released in the form of forming gas for safety reasons) through gypsum panel were obtained using an experimental apparatus, which was based on the 1/4-scale garage previously used for studying helium release and dispersion in our laboratory. Two types of gypsum panel were used in the experiments. Helium or forming gas was released into the enclosure

^v The estimated relative combined standard uncertainty of D_e was 8 % based on the standard uncertainties of the best-fit parameters, a and b , and the application of the law of propagation of uncertainty to all the parameters in Eq. (6) used to derive D_e .

^{vi} In this case, P denotes probability not pressure.

for a fixed duration and then terminated. The gas mixture in the enclosure was perfectly mixed, and the temporal and spatial gas concentrations were monitored using eight sensors located at different heights above the enclosure floor. The temporal variations of the pressure difference between the enclosure interior and the ambience were also measured. An analytical model was developed to extract the effective diffusion coefficients from the experimental data. The estimated average effective helium diffusion coefficients through regular and Type X gypsum panels were $1.4 \times 10^{-5} \text{ m}^2/\text{s}$ and $1.3 \times 10^{-5} \text{ m}^2/\text{s}$ respectively, and the average value for hydrogen through Type X gypsum panel was $1.4 \times 10^{-5} \text{ m}^2/\text{s}$.

Since the interior of most garages in the U.S. have large surface areas covered with gypsum panels together with the fact that hydrogen can readily diffuse through gypsum panels, this diffusion process should not be overlooked in the hazard assessment of accidental release of hydrogen in garages or enclosures lined with gypsum panels.

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Table 1. Helium effective diffusion coefficient through gypsum.

| Gypsum type | \dot{n}_f (mol/s) | D_e (m ² /s) from <i>a</i> | D_e (m ² /s) from <i>b</i> | $[D_e]$ (m ² /s) |
|--|-----------------------|---|---|-----------------------------|
| Regular* | 1.02×10^{-2} | 1.4×10^{-5} | 1.4×10^{-5} | 1.4×10^{-5} |
| Regular | 1.02×10^{-2} | 1.4×10^{-5} | 1.4×10^{-5} | 1.4×10^{-5} |
| Regular | 2.43×10^{-3} | 1.1×10^{-5} | 1.1×10^{-5} | 1.1×10^{-5} |
| Regular | 2.43×10^{-3} | 1.2×10^{-5} | 1.2×10^{-5} | 1.2×10^{-5} |
| Regular | 1.02×10^{-2} | 1.3×10^{-5} | 1.4×10^{-5} | 1.4×10^{-5} |
| Regular | 1.02×10^{-2} | 1.4×10^{-5} | 1.4×10^{-5} | 1.4×10^{-5} |
| Regular (painted) | 1.02×10^{-2} | 3.3×10^{-6} | 3.4×10^{-6} | 3.4×10^{-6} |
| Type X** | 1.02×10^{-2} | 1.2×10^{-5} | 1.3×10^{-5} | 1.3×10^{-5} |
| Type X | 1.02×10^{-2} | 1.3×10^{-5} | 1.3×10^{-5} | 1.3×10^{-5} |
| Type X | 1.02×10^{-2} | 1.2×10^{-5} | 1.3×10^{-5} | 1.3×10^{-5} |
| Type X | 1.02×10^{-2} | 1.3×10^{-5} | 1.3×10^{-5} | 1.3×10^{-5} |
| Type X | 1.02×10^{-2} | 1.3×10^{-5} | 1.3×10^{-5} | 1.3×10^{-5} |
| Type X | 1.02×10^{-2} | 1.3×10^{-5} | 1.3×10^{-5} | 1.3×10^{-5} |
| Type X | 1.02×10^{-2} | 1.3×10^{-5} | 1.3×10^{-5} | 1.3×10^{-5} |
| Type X | 1.02×10^{-2} | 1.2×10^{-5} | 1.3×10^{-5} | 1.3×10^{-5} |
| *SHEETROCK® Brand; $\delta = 0.0127$ m | | | | |
| ** $\delta = 0.0159$ m | | | | |

Table 2. Hydrogen effective diffusion coefficient through gypsum.

| Gypsum type | \dot{n}_f (mol/s) | D_e (m ² /s) from <i>a</i> | D_e (m ² /s) from <i>b</i> | $[D_e]$ (m ² /s) |
|-------------|-----------------------|---|---|-----------------------------|
| Type X | 3.09×10^{-4} | 1.6×10^{-5} | 1.4×10^{-5} | 1.5×10^{-5} |
| Type X | 3.09×10^{-4} | 1.5×10^{-5} | 1.2×10^{-5} | 1.4×10^{-5} |

Table 3. Effective diffusion coefficients of various gases through gypsum.

| Gypsum type | Gas | T (°C) | RH [§] (%) | D_e (m ² /s) | Reference |
|--|--------------|----------|---------------------|---------------------------|--------------|
| Regular & Type X | Helium | 22 | --- | 1.3×10^{-5} | Current work |
| Type X | Hydrogen | 22 | --- | 1.4×10^{-5} | Current work |
| Calcium silicate* | Formaldehyde | 23 | 25 | 3.08×10^{-6} | [9] |
| Calcium silicate | Formaldehyde | 23 | 50 | 3.29×10^{-6} | [9] |
| Calcium silicate | Toluene | 23 | 25 | 1.73×10^{-6} | [9] |
| Calcium silicate | Toluene | 23 | 50 | 1.72×10^{-6} | [9] |
| Calcium silicate | Water vapor | 23 | 50** | 3.04×10^{-6} | [9] |
| Calcium silicate | Water vapor | 23 | 80** | 3.06×10^{-6} | [9] |
| Unspecified | Water vapor | 23 | 5 | 4.1×10^{-6} | [12] |
| Unspecified | Decane | 23 | 5 | 2.8×10^{-6} | [12] |
| [§] Relative humidity *Simulated gypsum **nominal values in one chamber (dual-chamber method) | | | | | |

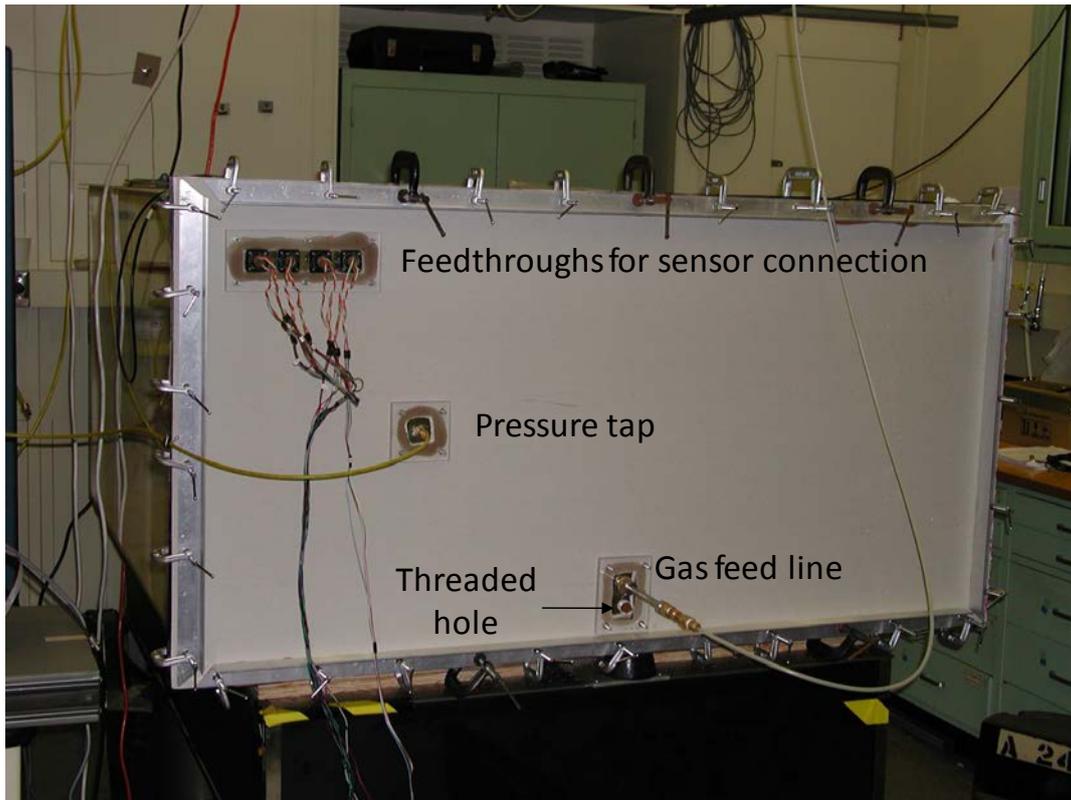


Figure 1. A photograph of the experimental apparatus.

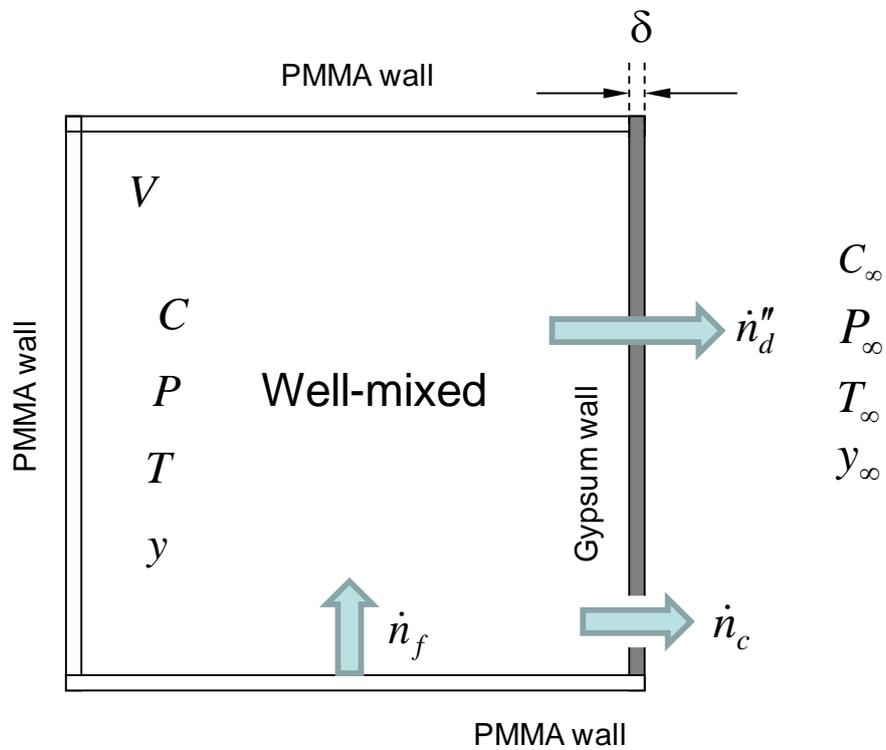


Figure 2. Schematic used for the analysis.

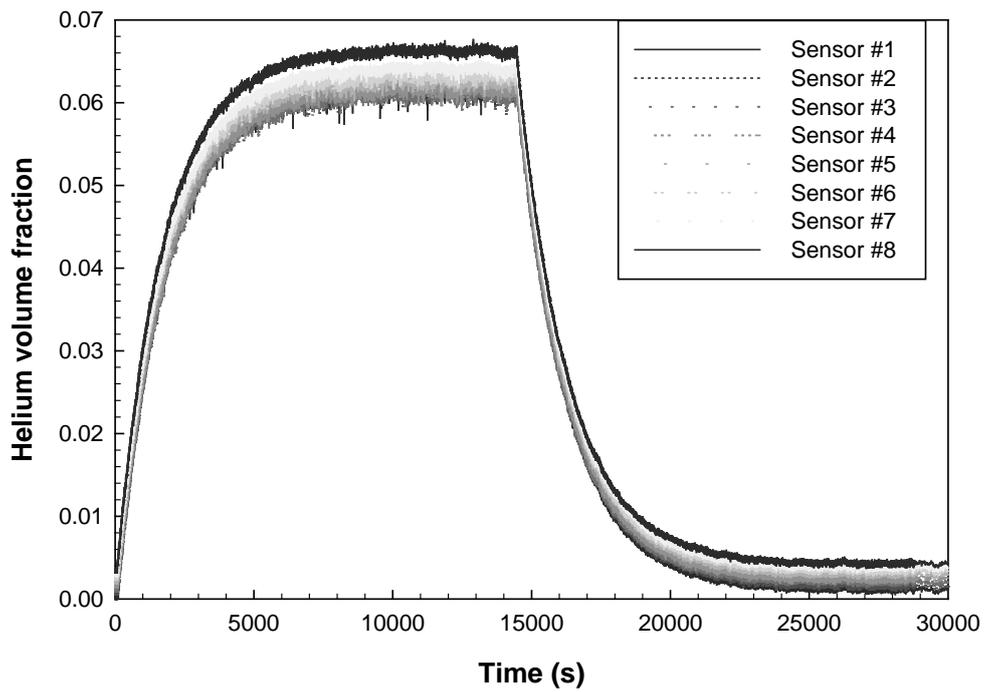


Figure 3. Helium concentration measurements from the eight sensors with $\dot{n}_f = 2.43 \times 10^{-3}$ mol/s.

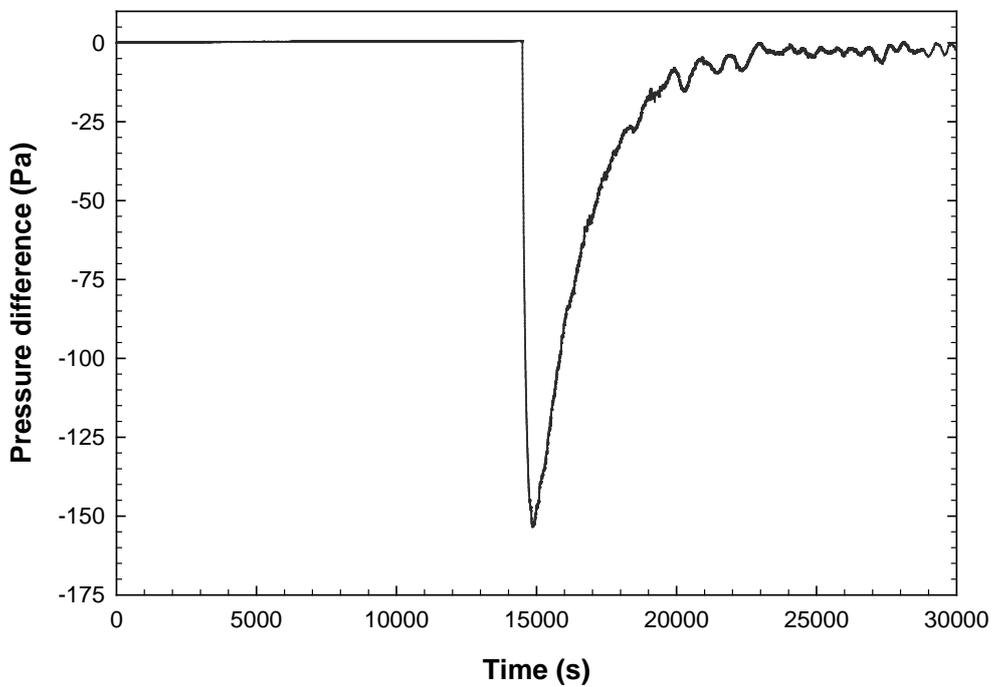


Figure 4. Temporal variation of measured $P - P_\infty$ with $\dot{n}_f = 2.43 \times 10^{-3}$ mol/s of helium.

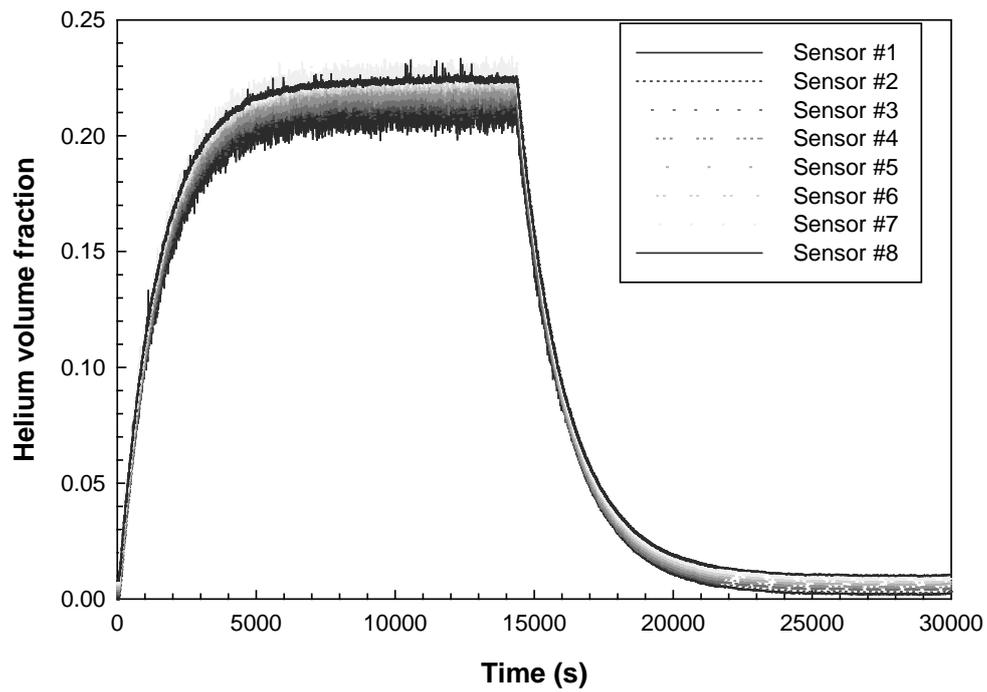


Figure 5. Helium concentration measurements from the eight sensors with $\dot{n}_f = 1.02 \times 10^{-2}$ mol/s.

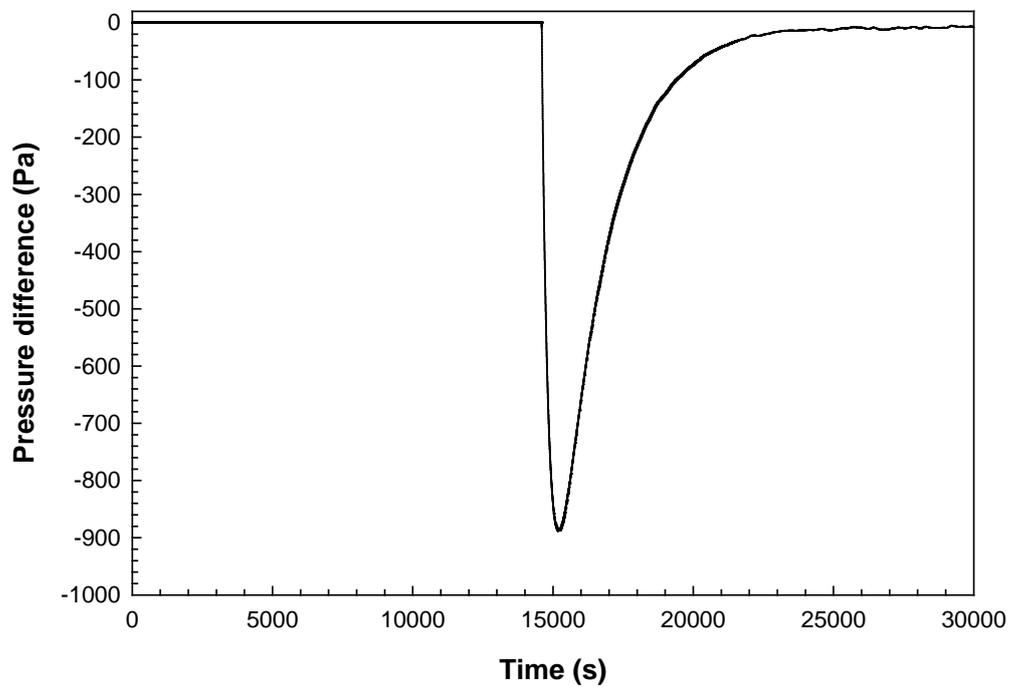


Figure 6. Temporal variation of $P - P_\infty$ with $\dot{n}_f = 1.02 \times 10^{-2}$ mol/s of helium.

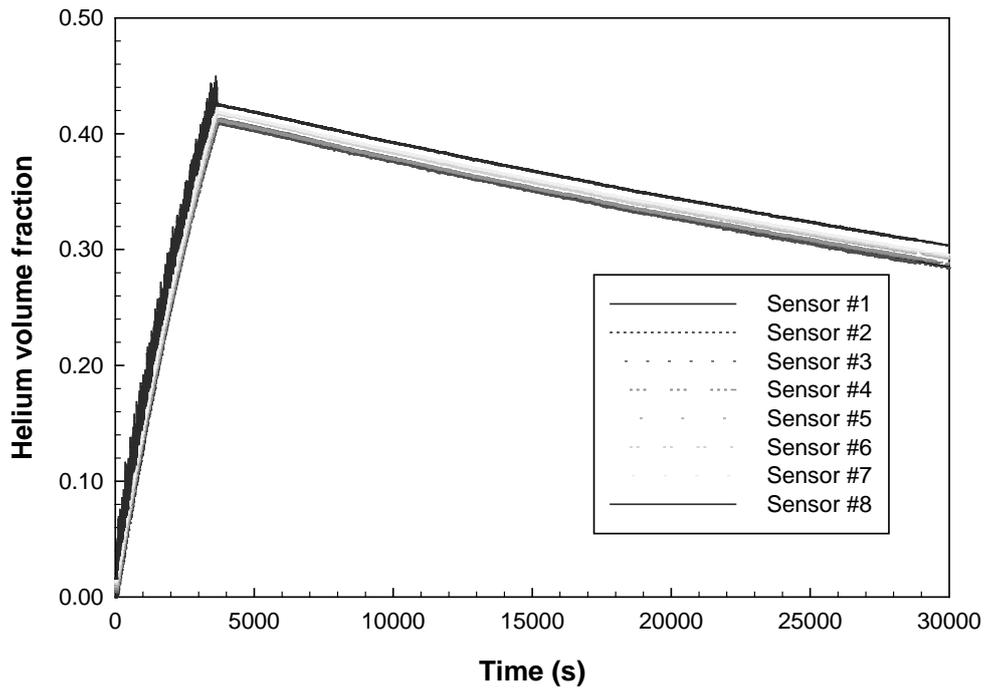


Figure 7. Helium concentration measurements from the eight sensors with $\dot{n}_f = 1.02 \times 10^{-2}$ mol/s (painted gypsum panel).

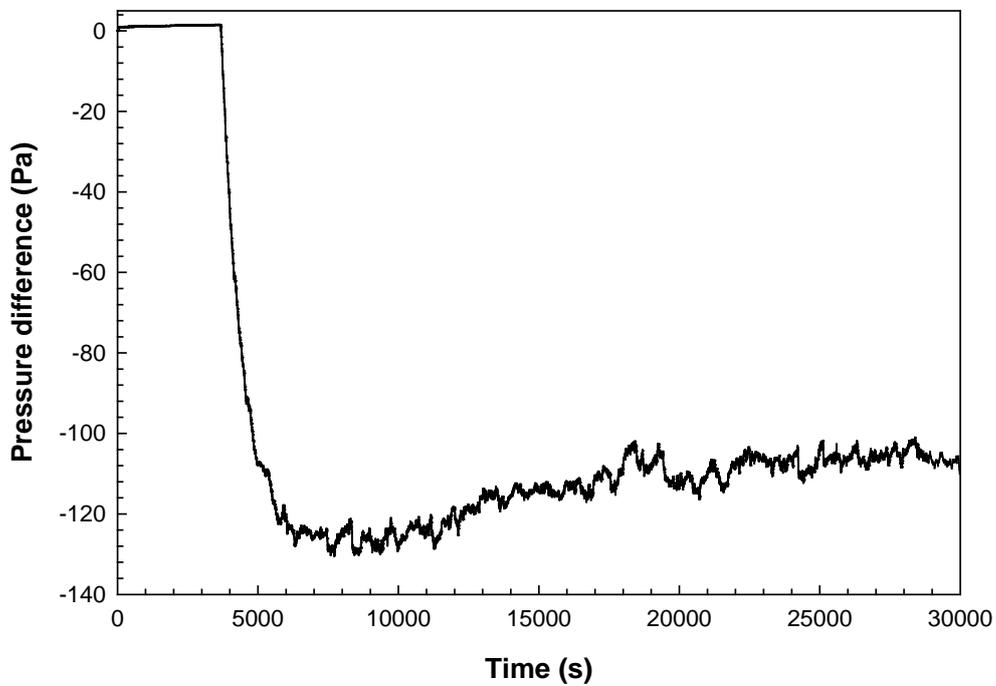


Figure 8. Temporal variation of $P - P_\infty$ with $\dot{n}_f = 1.02 \times 10^{-2}$ mol/s of helium (painted gypsum panel).

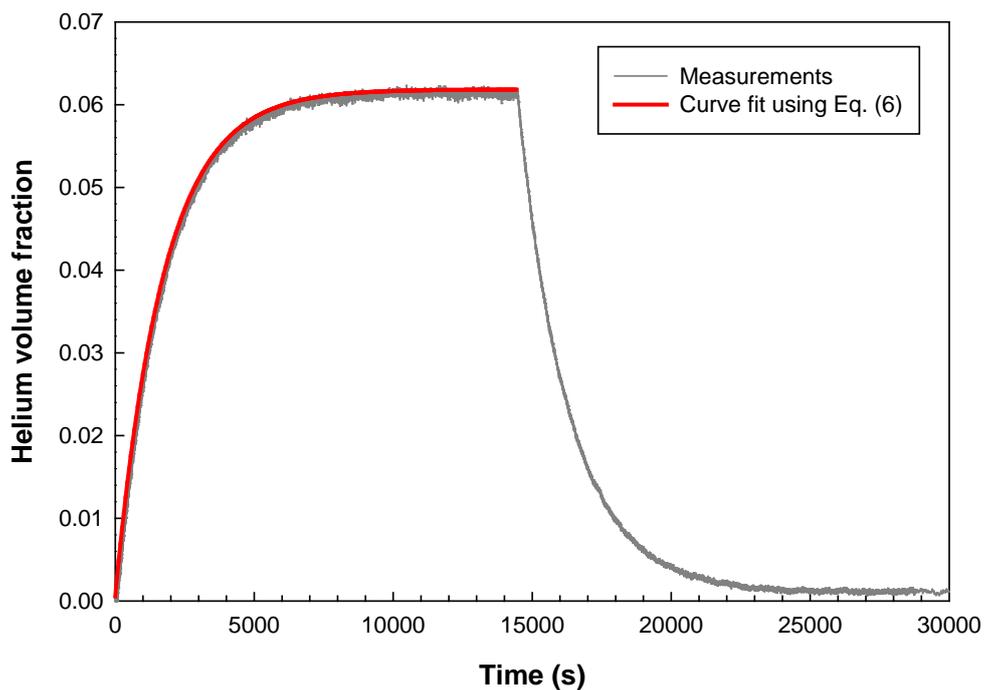


Figure 9. Curve fit in the form of Eq. (6) using the data (averages of eight sensor readings) in Fig. 3 to extract D_e .

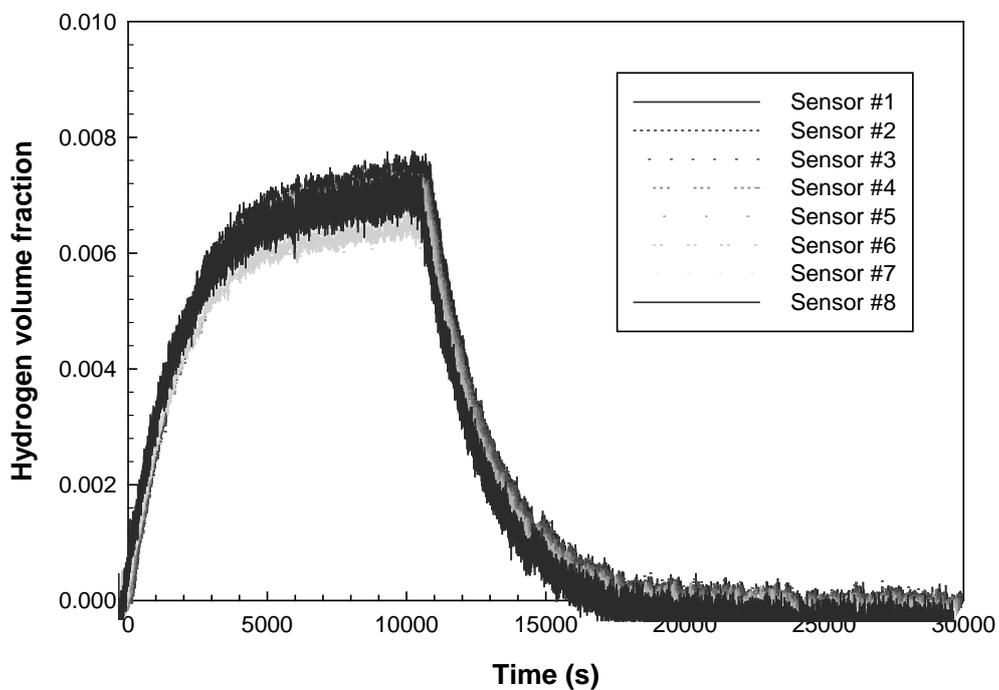


Figure 10. Hydrogen concentration measurements from the eight sensors with $\dot{n}_f = 3.09 \times 10^{-4}$ mol/s.

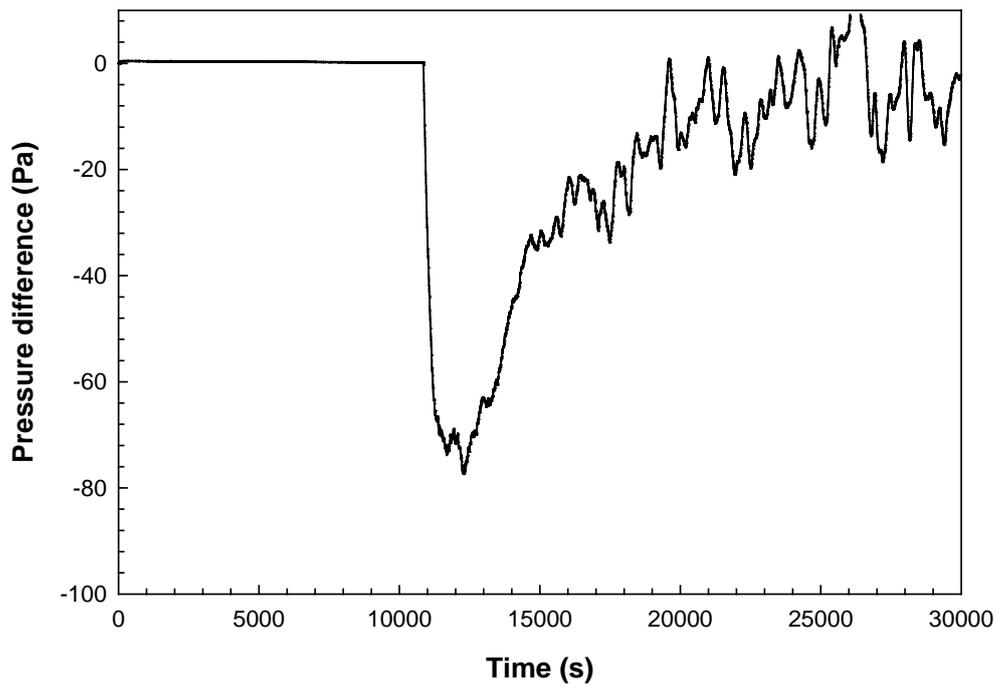


Figure 11. Temporal variation of $P - P_{\infty}$ with $\dot{n}_f = 3.09 \times 10^{-4}$ mol/s of hydrogen.