





# Multichannel gas-uptake/evolution reactor for monitoring liquid-phase chemical reactions

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Chase A. Salazar,  Blaise J. Thompson,  Spring M. M. Knapp,  Steven R. Myers, and Shannon S. Stahl<sup>a)</sup> 

## AFFILIATIONS

Department of Chemistry, University of Wisconsin-Madison, Madison, Wisconsin 53719, USA

<sup>a)</sup> Author to whom correspondence should be addressed: [stahl@chem.wisc.edu](mailto:stahl@chem.wisc.edu)

## ABSTRACT

The design of a headspace pressure-monitoring reactor for measuring the uptake/evolution of gas in gas-liquid chemical transformations is described. The reactor features a parallel setup with ten-reactor cells, each featuring a low working volume of 0.2–2 ml, a pressure capacity from 0 to 150 PSia, and a high sensitivity pressure transducer. The reactor cells are composed of commercially available disposable thick-walled glassware and compact monolithic weld assemblies. The software interface controls the reactor temperature while monitoring pressure in each of the parallel reactor cells. Reactions are easy to set up and yield high-density gas uptake/evolution data. This instrument is especially well suited to acquire quantitative time-course data for reactions with small quantities of gas consumed or produced.

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## I. INTRODUCTION

Liquid-phase chemical reactions that use gaseous reagents (e.g., O<sub>2</sub>, H<sub>2</sub>, CO, ethylene, and CO<sub>2</sub>) play an important role in laboratory and industrial chemical syntheses. Analysis of reaction kinetics is crucial for the development of these processes; however, quantitative data acquisition can be complicated by perturbation of the reaction mixture when sampling from a sealed system (e.g., with-drawing aliquots) or due to poor gas-liquid mixing [e.g., during nuclear magnetic resonance (NMR) spectroscopic analysis]. Monitoring the gas consumption or evolution provides an appealing non-intrusive method to bypass these limitations, and it has been successfully used to analyze diverse gas-liquid chemical reactions, including hydrogenations,<sup>1</sup> artificial photosynthesis,<sup>2</sup> carbonylations,<sup>3</sup> ethylene polymerization,<sup>4</sup> ammonia borane dehydrogenation,<sup>5</sup> aerobic oxidations,<sup>6</sup> and hydroformylations.<sup>7</sup>

Monitoring reaction headspace pressure is especially useful in quantifying consumption or evolution of small quantities of gas. For example, liquid-phase catalytic aerobic oxidation reactions, such as those recently investigated in our lab and by others,<sup>8,9</sup> utilize relatively dilute reaction concentrations (50–300 mM) and commonly consume only 0.5 equivalents of O<sub>2</sub>, resulting in relatively small quantities of gas consumed. Many commercially available reactors

are available, and these are typically designed to accommodate high gas pressures (e.g., 1500–3000 PSia). The large dynamic range of these systems leads to a low resolving power for analysis of reactions that consume small amounts of gas.<sup>1,7,10,11</sup>

In the present report, we describe a low-volume, multi-channel reactor system for parallel analysis of headspace pressure. It is compatible with relatively low pressures and capable of analyzing reactions that consume or evolve small quantities of gas. The reactor described here complements previously reported reactor systems for analysis of small-scale gas uptake/evolution reactions based on the measurement of gas flow rates,<sup>12,13</sup> differential pressures,<sup>14</sup> bubble counts,<sup>15</sup> and headspace volume<sup>16</sup> or pressure<sup>2,6,10,11</sup> changes. Key criteria incorporated into the present reactor design were not collectively incorporated into previously reported systems: (1) compatible with pressure ranges of 0–150 PSia; (2) high sensitivity, allowing for the quantitative detection of gas uptake/evolution in amounts of  $\leq 0.2 \mu\text{mol}$ ; (3) small reaction volumes (0.2–2 ml) that minimize reagent quantities need to analyze the reactions; (4) compatible with disposable reaction vessels that facilitate setup, minimize gas leakage, and enhance user-friendly operation by non-specialists; (5) a compact, parallel design to accommodate up to ten simultaneous reactions; (6) allow for safe analysis of aerobic oxidation reactions, with little or no potential for fire propagation and/or explosion;