



## Characterization and Automation of Hydrazine Detector Calibrations for the United States Air Force

### Army Issues and Technology Impact

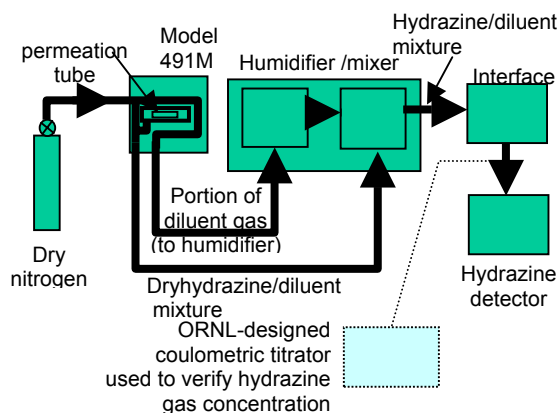
Hydrazine ( $\text{N}_2\text{H}_4$ ) is a highly toxic liquid used as an aerospace fuel, an antioxidant in industrial processes, and in the production of pesticides and pharmaceuticals. Hydrazine vapor in air is flammable at 4.7 to 100% hydrazine by volume. The personnel exposure limit (Part 1910 of Title 29 of the Code of Federal Regulations (29 CFR 1910.1000)) is 1 ppm ( $1.3 \text{ mg/m}^3$ ). The American Conference of Governmental Industrial Hygienists (ACGIH) (1997) has recommended 10 ppb ( $0.013 \text{ mg/m}^3$ ) as the threshold limit value for occupational exposures to hydrazine in workplace air.

Hydrazine is used extensively by the United States Air Force and, because of the stringent exposure requirements, there is a great need for developing improved calibration methods (i.e., lower uncertainty, reduced calibration time, and faster turn-around time). ORNL has developed such a method for calibration of hydrazine monitors in the Air Force inventory. Calibration uncertainty has been reduced by 50%, calibration time has been reduced by more than 20%, and the cost per calibration has also been reduced. In addition, the effect of humidity on hydrazine calibrations has been characterized.

### Technical Concept

The hydrazine detector calibration system is composed of a Kintek 491 reference gas generation system, a humidifier/mixer system which combines the dry reference hydrazine gas with humidified diluent or carrier gas to generate the required humidified reference for calibrations, and a gas sampling interface.

The Kintek reference gas generation system is calibrated using an ORNL-designed coulometric titration system to verify the hydrazine concentration of the sample atmosphere in the interface module. The sample atmosphere is sampled by pumping a measured flow of the reference gas into an impinger for a measured time interval at the measured temperature and pressure. The amount of hydrazine in each of these impinger samples is then determined using a coulometric titration procedure.



ORNL method for calibrating hydrazine monitors in the Air Force inventory

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Using the results of the concentrations determined by the coulometric titration a calibration equation is created for the Kintek 491 reference gas generation system. This calibration equation is then used to determine the concentration of the reference gas in the sample atmosphere of the interface module. The test instrument (hydrazine monitor) is then connected to the sample atmosphere and calibrated based on the sample atmosphere's known concentration as determined by the Kintek 491 reference gas generation system's calibration equation. Note that collection of impinger samples, with subsequent coulometric titration, is not needed every time to calibrate the hydrazine monitors. It is only necessary to collect impinger samples each time the Kintek 491 reference gas generation system is calibrated. Once calibrated, the Kintek 491 reference gas system can be used to calibrate several hydrazine monitors for several months before it may need to be recalibrated.

Using this calibration method, calibration uncertainty has been reduced by 50% compared to the current method used to calibrate the hydrazine monitors in the Air Force inventory. Calibration time has also been reduced by more than 20%, and the cost per calibration has been reduced. In addition, the effect of humidity on hydrazine calibrations has been characterized.



**ORNL-designed coulometer circuit board.**

Phase 1 of the project, which involved the development of the calibration method, has been completed. ORNL is currently working on Phase 2 of the project, which involves the development of a web-based Calibration procedure that Air Force personnel can use to calibrate hydrazine monitors in-house.

#### **ORNL Facilities**

ORNL has strong capabilities in metrology, electronic design chemical analysis, and measurement science. The Metrology Services and Measurement Standards Laboratory (MS&MS) supplies metrology and testing capabilities for ORNL R&D and support organizations. The facility provides the expertise and resources for the conduct of a wide variety of tests and measurements, and represents the top level of traceability for physical (pressure, flow, vacuum, temperature) and electrical measurement parameters at ORNL. This project combines metrology, electronic design, and chemical analysis expertise available at ORNL.

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