Ensuring Oxygen-Free Conditions in Thermogravimetric Analysis

1. Background

Maintaining an inert, oxygen-free atmosphere is essential in many thermogravimetric analysis (TGA) applications, particularly when studying materials that are sensitive to oxidation at elevated temperatures. While most commercial TGA instruments provide adequate oxygen exclusion for routine polymer and hydrocarbon analyses, more demanding applications—such as oxidation-sensitive metals, catalysts, and carbonaceous materials—require significantly tighter control of residual oxygen levels.

This application note describes practical methods for evaluating purge gas effectiveness and oxygen exclusion in TGA instruments. It also highlights how **AMI's** TGA design achieves excellent oxygen removal efficiently, without reliance on excessive purge flow rates or complex gas-control hardware.

The acceptable level of residual oxygen depends strongly on the experiment. Routine polymer and hydrocarbon analysis tolerates trace oxygen without measurable impact. Carbon materials, finely divided metals, and catalysts may oxidize in the presence of very small amounts of oxygen. Kinetic and long isothermal experiments are particularly sensitive, as even slow oxidation rates become detectable over time.

Rather than assuming performance, purge effectiveness should be verified experimentally using sensitive indicator materials and well-designed test protocols.

2. Practical Methods for Evaluating Oxygen Presence

2.1 Carbon Black (Activated Charcoal)

Carbon black provides a simple, inexpensive, and sensitive test for residual oxygen. At temperatures ≥700 °C, carbon black oxidizes to CO_2 in the presence of oxygen, resulting in measurable mass loss.

- ✓ Method: An isothermal hold at high temperature under inert purge.
- ✓ Performance goal: ≤0.1 µg/min mass loss indicates excellent oxygen exclusion in well-sealed systems.

Older carbon black samples are preferred, as highly reactive surface sites are passivated, improving repeatability. Any crucible material may be used. Detectable mass loss at the microgram-per-minute level typically indicates air leakage, insufficient purging, or permeation through tubing.
Carbon black tests are also useful for determining required purge times prior to analysis, effectiveness of higher initial purge rates, and impact of furnace opening between runs.

2.2 Oxidizable Metals and Copper Oxalate

For more stringent oxygen detection, oxidizable metals such as titanium, zirconium, or tungsten powders may be used, though these materials present inhalation and explosion hazards.
A safer and highly sensitive alternative is copper oxalate. Copper oxalate decomposes near 325 °C, producing freshly reduced, finely divided copper. The high surface area and fresh metal surface make it extremely sensitive to trace oxygen. Oxidation manifests as weight gain or color change (gray to white).

- ✓ Method: An isothermal hold ~1000 °C under inert purge.
- ✓ Performance goal: Residue should remain gray with minimal weight change after initial decomposition around 325 °C.

Copper oxalate hemihydrate is commercially available and offers improved reproducibility compared to loose carbon materials.

3. Importance of Purge Strategy and Gas Delivery

3.1 High Initial Flow Rates

High initial purge rates help rapidly displace trapped air, especially after opening the furnace or switching from oxidizing to inert atmospheres. Flow rates up to 300 mL/min for 15–30 minutes may be required in large furnace volumes. Longer purge times may be necessary in systems with significant dead volume or gas path complexity. Opening the furnace only when necessary can significantly reduce re-purge time.

3.2 Leak Prevention and Gas Purity

Persistent oxygen signals often originate from sources other than external air leakage. Polymer tubing is oxygen-permeable and should be replaced with clean stainless-steel tubing for critical work. Copper tubing should be avoided due to surface oxidation. Properly seated O-rings, fittings, and gas connectors are essential. High-purity inert gases are recommended; inline heated gas purifiers may further reduce residual oxygen. Lastly, some ceramics, such as alumina, can absorb and release oxygen during heating and cooling and should be avoided when ultra-low oxygen levels are required.

4. AMI TGA Design Advantage: Micro-Furnace with Dual-Zone Purge

Traditional TGA systems often rely on high purge flows and mass flow controllers (MFCs) to overcome large furnace volumes and inefficient gas paths. In contrast, **AMI** TGA instruments employ a fundamentally different approach.

<u>Micro-Furnace Architecture</u>

- ✓ Compact furnace design minimizes dead volume.
- ✓ Short gas paths reduce oxygen residence time.
- ✓ Rapid establishment of inert conditions is achieved even at low flow rates.

Dual-Zone Purge System

- ✓ Sample zone purge directly surrounds the crucible, ensuring oxygen is displaced precisely where the reaction occurs.
- ✓ Balance zone purge protects the microbalance from reactive gases, vapors, and thermal disturbances.

This architecture provides more effective oxygen exclusion than single-zone, high-flow systems.

5. Case Study

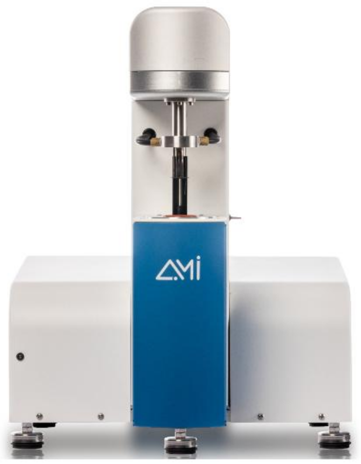
A carbon black oxidation test was used to validate **AMI** purge performance. About 10 mg of carbon black was loaded into the **TGA 1000**, and the sample was held isothermally at 700 °C under flowing nitrogen (50 mL/min).

The carbon black sample resulted in less than 1% mass loss over 30 minutes. These results confirm stable oxygen-free conditions under thermal stress without high purge rates or MFCs.

6. Conclusions

Achieving oxygen-free conditions in TGA depends on both purge strategy and instrument design. While high flow rates and elaborate gas controls can reduce oxygen levels, **AMI** TGA systems demonstrate that superior oxygen exclusion is best achieved through efficient furnace geometry and targeted dual-zone purging. In many systems, high flow rates compensate for inefficient furnace design—but at increased cost and complexity. The **AMI** approach demonstrates that:

- ✓ Minimizing furnace volume is more effective than brute-force gas flushing
- ✓ Dual-zone purge design outperforms single-zone, high-flow systems
- ✓ Eliminating MFCs reduces maintenance, cost, and system failure points without sacrificing performance



TGA 1000

- ✓ Research-grade capabilities at an accessible price point
- ✓ High sensitivity microbalance
- ✓ Resolution down to 0.1 µg
- ✓ Compact heating furnace
- ✓ Precise temperature control

Figure 1: Highlighted features of ***TGA 1000*** *from* ***AMI***

With its micro-furnace architecture and optimized gas flow design, the **AMI TGA 1000** delivers excellent purge effectiveness at low gas flow rates, reliable performance for oxygen-sensitive materials, and reduced operational complexity and cost. These capabilities make **AMI** TGA systems a practical and powerful solution for laboratories requiring dependable inert-atmosphere thermogravimetric analysis.