TA Instruments

Thermal Analysis & Rheology

# THERMAL SOLUTIONS

## DETERMINATION OF OIL IN RUBBER BY VACUUM TGA

### PROBLEM

Many rubber products are made of simple mixtures of oil, raw rubber, and carbon black. End-use performance of these materials is dependent on batch composition, with variations of even 1 or 2% causing significant differences. For this reason, manufacturers of tires, seals, gaskets, and vibration-damping mounts require a way to quickly assess batch composition.

Thermogravimetric analysis (TGA), which is a thermal analysis technique used to measure weight changes in a material with temperature, is commonly used to determine the composition of multicomponent rubber products. However, conventional TGA (flowing atmosphere) cannot always separate and quantify the oil content since the rubber present begins to pyrolyze before all of the oil evaporates.

### SOLUTION

Separation of weight losses associated with evaporating oils and decomposing rubber can be resolved by running TGA under vacuum. The Simultaneous TGA-DTA (or SDT) 2960, because of its horizontal design, is ideal for vacuum work. The SDT is easily modified for vacuum studies by fitting a vacuum connection to the end of the furnace tube and capping off the standard purge port.

Figure 1 shows the oil and rubber weight losses for a commercial rubber under a nitrogen purge. Figure 2 shows the TGA results on the same material performed under a reduced atmosphere of 5 Pa (0.04 mm Hg). Improved resolution between the overlapping weight losses is obtained because the weight loss associated with oil evolution is moved to lower temperatures under vacuum. Although lower pressures produce the best results, even moderate levels of vacuum can improve the resolution as demonstrated by the parent weight loss and derivative curves in Figures 3 and 4 respectively.

Figure 1. NORMAL TGA AT AMBIENT PRESSURE

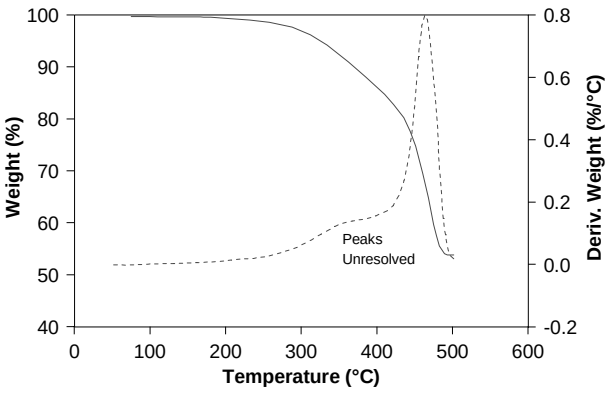



Figure 2. TGA AT 5Pa

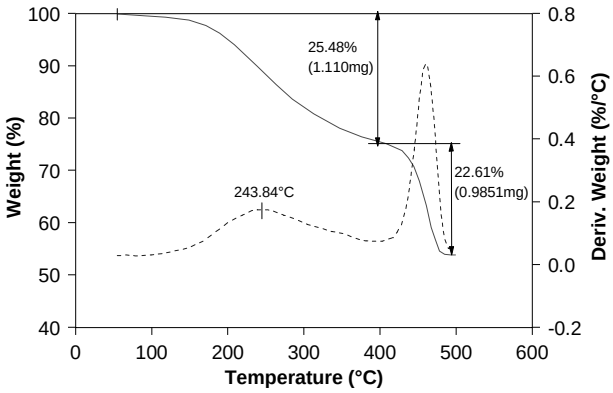


**Figure 3. TGA AT INCREASING PRESSURES**

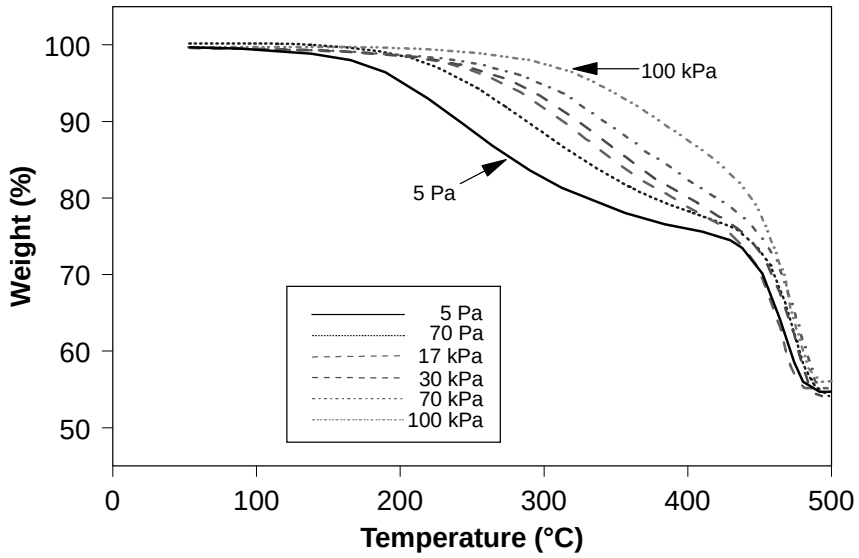
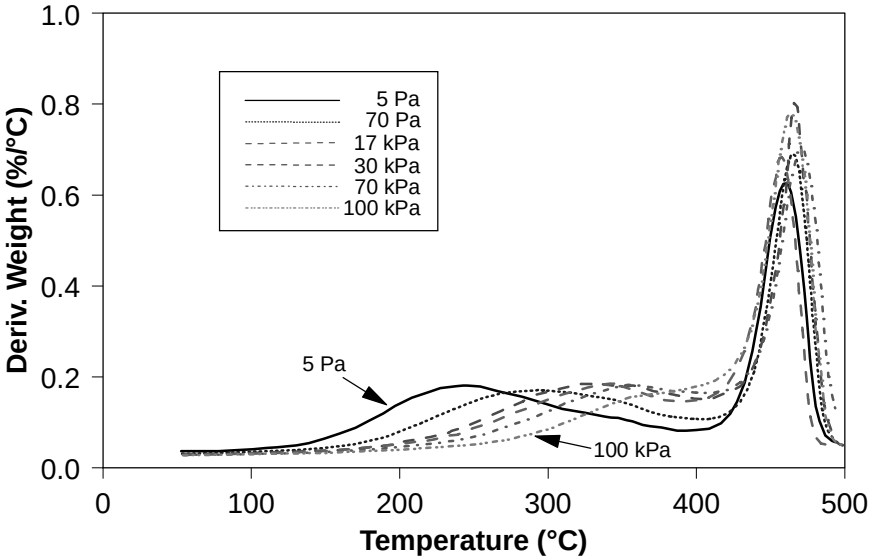



**Figure 4. DERIVATIVE TGA AT INCREASING PRESSURES**




**Acknowledgment:** This brief is based on studies performed by Blaine Weddle and C. Jay Lundgren, TA Instruments Applications Laboratory (US).

For more information or to place an order, contact:
**TA Instruments, Inc.**, 109 Lukens Drive, New Castle, DE 19720, Telephone: (302) 427-4000, Fax: (302) 427-4001
**TA Instruments S.A.R.L.**, Paris, France, Telephone: 33-01-30489460, Fax: 33-01-30489451
**TA Instruments N.V./S.A.**, Gent, Belgium, Telephone: 32-9-220-79-89, Fax: 32-9-220-83-21
**TA Instruments GmbH**, Alzenau, Germany, Telephone: 49-6023-30044, Fax: 49-6023-30823
**TA Instruments, Ltd.**, Leatherhead, England, Telephone: 44-1-372-360363, Fax: 44-1-372-360135
**TA Instruments Japan K.K.**, Tokyo, Japan, Telephone: 813-5434-2771, Fax: 813-5434-2770

**Internet: http://www.tainst.com**



TA Instruments
Thermal Analysis & Rheology
A Subsidiary of Waters Corporation