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Quality control and the mixing process

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4.1 INTRODUCTION

Some of the factors that repeatedly enter into the mixing of rubber compounds are relatively straightforward: the raw materials used must be consistent; the same mix procedure must be followed, with the same heat and shear history throughout the process; contamination must be avoided. Other factors are less obvious, but nevertheless important: the compound should be formulated so as to minimize the effect of small variations in processing, and to minimize the chances for errors in composition. The type of mixing procedure (the order in which the ingredients are added) should be appropriate to the formulation of the ingredients and to the design of the internal mixer, so as to minimize the dependency of compound properties on small process variations. Finally, the mixer itself must be well maintained (clearances within specifications, proper circulation of coolant, etc.) for a consistent product to be obtained. This chapter will be concerned with measurement of the consistency of raw materials and finished compound.

4.2 TESTING OF RAW MATERIALS

Consistent mixed compound cannot be obtained without testing each lot of every raw material. Such a program is invariably expensive. The consequences of not testing inevitably prove to be more expensive. Every raw material should be examined visually. This will eliminate most cases
of contamination (except for inadvertent contamination caused later on). Merely looking at the ingredient before using it will identify some defects, such as polymers with oxidized surface layers. Many cases of mistaken identity (or mislabeling) will be similarly spotted. The raw material specification for every ingredient should include visual inspection.

Each lot of incoming raw material should include certified test data for that lot, signed by a responsible individual on behalf of the supplier. The certification should contain actual test data, not merely a statement that the shipment complies with the supplier's sales specifications (the latter often furnished with 'typical' test data). What is desired is specific data that can be used for statistical tracking, and repeated on checking. With many ingredients in routine use, the combination of visual examination and perusal of the batch certification will prove sufficient. Comparison of individual certifications with the published specification on the ingredient should not be ignored; many cases will be found where lots deviating from the supplier's specification have been inadvertently (or otherwise) shipped. Judgment will be needed in such cases to decide whether a shipment must be returned, or may be used (perhaps in one compound, but not in another).

4.2.1 Elastomers as raw materials

Incoming polymers should have their certification carefully checked for values of ash and volatiles. Unusually high values should be questioned. High ash should not reflect incursions of dirt or grit. High levels of volatiles may correlate with a moisture content great enough to make filler incorporation difficult. If in doubt, sample the polymer and run a test batch in the laboratory under close observation.

In cases where compound processability is critical, visual inspection and audit of certified data should be supplemented by a check of polymer viscosity. It is usually the Mooney viscosity that is compared with the supplier's data. For this to be meaningful, it must be established that exactly the same test procedure is used. ASTM D1646 (Mooney viscosity of natural and synthetic rubbers) includes several variations. It is necessary to use exactly the same sample preparation to duplicate results. And the test instruments should be calibrated by analogous methods. Some discrepancies have actually been traced to use of the wrong sized rotor. More often the lack of correlation stems from a worn rotor or worn dies, inadequate temperature control, and variation in sample preparation. A former source of variation, die spacing, has been minimized by modern design, but will still affect older, hand-closed Mooney viscometers. Other viscometers or plastometers can also be used; in fact, extrusion plastometers are more appropriate for