
Materiali, pridobljeni iz izrabljenih avtomobilskih gum - Vonj granulatov ELT - Vir in možnosti sanacije

Materials obtained from End-of-Life Tyres - Odour of ELT granulates - Origin and remediation possibilities

Materialien aus Altreifen - Geruch von ELT-Granulaten - Ursprungs- und Sanierungsmöglichkeiten

Matériaux produits à partir de pneus usagés non réutilisables (PUNR) - Odeur des granulats - Origine et possibilités de remédiation

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ICS

English Version

Materials obtained from End-of-Life Tyres - Odour of ELT
granulates - Origin and remediation possibilities

Materialien aus Altreifen - Geruch von ELT-Granulaten
- Ursprungs- und Sanierungsmöglichkeiten

This draft Technical Report is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 366.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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FprCEN/TR 17511:2020 (E)

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European foreword

This document (FprCEN/TR 17511:2020) has been prepared by Technical Committee CEN/TC 366 “Materials obtained from End-of-Life Tyres (ELT)”, the secretariat of which is held by UNI.

This document is currently submitted to the Vote on TR.

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FprCEN/TR 17511:2020 (E)**Introduction**

The odour of ELTs granulates can be an obstacle to their use. This is particularly critical at high temperatures, e.g. in the case of a plastic injection.

In order to solve this problem, the origin of the odour is identified to the extent that this is possible. Then it is quantified under normal conditions of use or at higher temperatures.

The identification of the exact origin of the odour would allow the possibility of reducing or eliminating it.

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1 Scope

The purpose of this document is to provide a review of the studies that were performed on odour of ELT granulates.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 14243-1, *Materials obtained from end of life tyres — Part 1: General definitions related to the methods for determining their dimension(s) and impurities*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 14243-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <http://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.2 Symbols and abbreviated terms

For the purposes of this part, the following symbols apply.

| | |
|-----|---------------------------|
| VOC | Volatile Organic Compound |
| NR | Natural Rubber |

4 Determination of the intensity

4.1 Methods

In a first study [3], granulates from six different suppliers were used. The techniques that were assessed for the determination of the intensity of the odour were the following:

- **Renault D49-3001:** this is one of the standardized tests for odour and smell that are commonly used by the automotive industry. In this method, the samples in solid form are placed in a sealed container between 70 °C and 100 °C during 2 h. A jury of at least five trained panellists then gives an intensity on a scale from 1 to 5. The nature of the smell is then placed in a family. The scale is defined as:
 - 0: no perceptible odour;
 - 1: weak odour, demanding extra attention and hard to describe;
 - 2: the subject perceives by simple smelling, without any other information;
 - 3: odour perceived even when the attention of the subject is elsewhere;
 - 4: powerful odour attracting the attention of the subject and hindering his other activities;
 - 5: unavoidable odour, focusing the attention of the subject.

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- **Dynamic olfactometry:** this is also a human-based method. The odour is diluted with a neutral gas until it is not detected by the panellist anymore. This gives a threshold concentration.
- **Hedonic test:** this is also a human-based method, to assess if the odour is pleasant or not.
- **GC-MS olfaction test:** this is a mixed human/machine method. It couples traditional gas chromatographic analysis with sensory detection in order to study complex mixtures of odorous substances and the molecules that are responsible for the smell.

The first observation is that there is no clear correlation between the Renault and the dynamic olfactometry methods. The second point is that no correlation between GC peaks and odour intensity could be made, probably because of synergy effects.

Given that the priority is the lowering of the smell intensity which is a subjective indicator, a human based method seemed to be the most relevant. Considering these results, Aliapur introduced a French standard (XP T47-766) following a procedure close to Renault D49 3001. Indeed, the preparation of the sample is crucial and shall be always the same if intensities are to be compared properly.

5 Origin of the odour

5.1 General

Following the results of this first study, two other studies were led to understand the origin of the odour perceived by users, in two different configurations: synthetic turfs and elastomer thermoplastic compounding. Temperatures are different in both cases, and so are the mechanisms at the origin of the odour.

5.2 At room temperature

5.2.1 Description of the study

A study [4] was performed to evaluate the odour of ELT granulates at a temperature of 55 °C and 65 °C, which roughly corresponds to the maximum temperatures that are achievable on an outside synthetic turf. Granulates and reference vulcanized rubber slabs were placed at a temperature of 55 °C or 65 °C before the test.

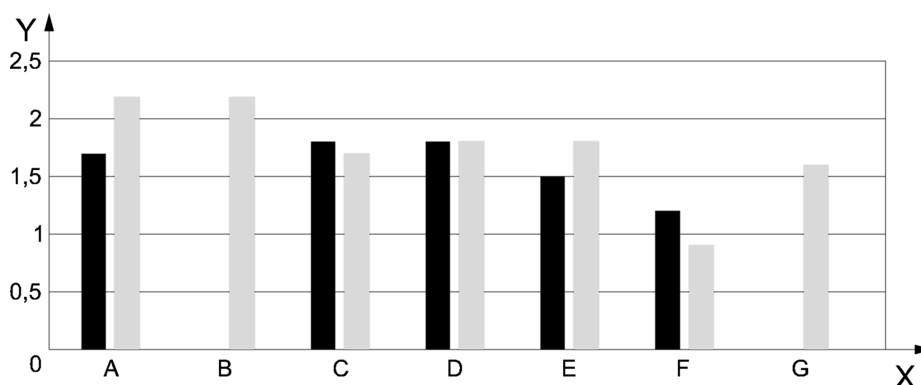
The odour was then evaluated (intensity and nature) by using Renault D49 3001 standard. Seven samples with different granulometries and from different production sites were tested.

Table 1 — Granulates tested in study [4]

| Sample ID | Origin | Granulometry |
|---|---------------|--------------|
| A | Passenger car | 0–0,5 mm |
| B | Passenger car | 0,5–1,5 mm |
| C | Truck | 0,5–1,5 mm |
| D | Passenger car | 0,5–1,5 mm |
| E | Truck | 0,5–2 mm |
| F | Truck | 1–2 mm |
| G | Passenger car | 0,5–2,5 mm |
| NOTE Sample B and sample D are different samples. | | |

5.2.2 Measured intensities for granulates

The measured intensities are given in Figure 1. Variations of intensities between 55 °C and 65 °C are low, and within the error of measurement. Sample B and sample G were not measured at 55 °C.

**Key**

- Y intensity
 X sample
 ■ 55° C
 ■ 65° C

Figure 1 — Measured intensities at 55 °C and 65 °C [4]**5.2.3 Odour and molecular origin**

The odour is generally qualified as “rubber”, and for some samples other descriptors occur such as “acid”/“acid”. This acrid smell mostly comes from residues of accelerators, residues from antioxidants and aldehydes.

The study of a reference formula as shown in Table 2, based on natural rubber and polybutadiene and common additives (components of the sulfur vulcanization system, rubber antioxidant agents like 6PPD) found in tyres, allows the molecules released by the granulates at 65 °C to be connected to the various additives:

Table 2 — Additives responsible for odour in reference mixtures

| Molecules from NR | Molecules from 6PPD | Molecules from sulfur vulcanization system |
|--|---|--|
| <ul style="list-style-type: none"> — piperidinone — methyl indole (animal) | <ul style="list-style-type: none"> — aniline (acrid, amine) — methyl benzene amine — methylisobutyl ketone | <ul style="list-style-type: none"> — methyl pentanamine — cyclohexylamine (acrid) — cyclohexanone (peppermint) — isocyanatocyclohexane (acrid) — benzothiazole (aromatic) — cyclohexyl formamide (acid) — cyclohexyl acetamide — diphenyl ether (unpleasant) |

The following molecules, detected in raw NR, are not detected in ELT granulates:

- pentanal;
- methyl butanoic acid;
- pentanoic acid;
- methyl propanal (acrid).

The acrid/acid descriptors can be connected to the following molecules:

- cyclohexylamine (acrid, fish);