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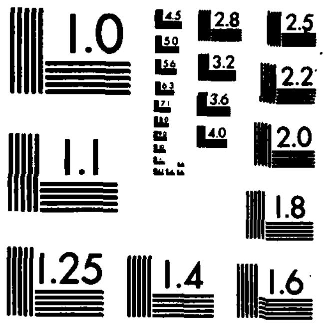

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DIRECT ANALYSIS OF HEAVILY LOADED CARBON BLACK FILLED STYRENE-BUTADIENE RUBBER USED IN TANK TRACK PADS BY FT-IR

JAMES M. SLOAN and JOSEPH P. CLEMENTS

POLYMER RESEARCH DIVISION

ARMY MATERIALS AND MECHANICS RESEARCH CENTER

June 1984

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ABSTRACT

Infrared spectra have been obtained of a heavily loaded (50 per hundred) carbon black filled styrene-butadiene rubber using a Fourier transform infrared spectrometer. Attenuated total reflectance (ATR) spectroscopy sampling employing a germanium (Ge) internal reflectance element was found to yield quality infrared spectra where other techniques have failed. Subtle changes were observed between the spectra of the filled and unfilled styrene-butadiene rubber (SBR). These changes are attributed to the interaction between polymer and filler.

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INTRODUCTION

Carbon black is the most widely used reinforcement agent presently in use in the elastomer industry. The role of carbon black in the reinforcement of elastomers is an important one. Addition of carbon black to an elastomeric system improves the mechanical properties of the final vulcanizate. Considerable improvement in tensile strength, tear resistance, and abrasion resistance has already been realized.[1] Such variables as the size and shape of carbon black particles, their dispersability and the chemical nature of their surfaces[1,2] contribute to the degree of reinforcement. Theories of reinforcement of elastomers by carbon black postulate the existence of a physical or chemical interaction at the polymer-filler interface.

Carbon black has also been found to increase the rate of vulcanization and improve reversion resistance.[3] The degree of improvement in reversion resistance is largely dependent on the size of the carbon black particles.

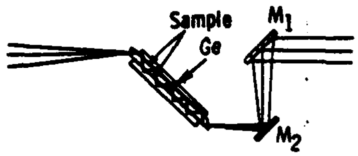
Typical industrial rubber formulations include anywhere from 40 to 60 parts carbon black per hundred parts elastomer. Obtaining infrared spectra of materials with these high loadings of carbon black is not an easy matter. Corish[4] obtained an IR spectrum of a heavily loaded carbon black filled styrene-butadiene rubber (SBR) using a microtomed (2-microns thick) slice aligned in the IR beam. Unfortunately, he used a grating spectrometer which allowed only a small fraction of radiation to reach the detector. This resulted in a spectrum of poor quality. A recent report[5] used the above method in conjunction with a Fourier transform infrared spectrometer with greatly improved results. Hart et al.[6] demonstrated the usefulness of using transmission sampling through dilution with KBr with an FT-IR spectrometer to obtain quality IR spectra of a 20 per hundred carbon black filled polybutadiene rubber.

The work presented in this paper demonstrates an easy method of sample preparation to obtain quality infrared spectra of heavily loaded (50 per hundred) carbon black filled SBR. The method utilizes an attenuated total reflectance (ATR) accessory employing Ge as the internal reflectance element (IRE). The quality of the IR spectrum is of sufficient quality to allow detection of small structural changes. These changes are thought to be a result of the interaction between the rubber and the carbon black filler.

EXPERIMENTAL

All spectra reported were recorded using a Harrick Model 4X-TBC-VA ATR accessory. The IRE was a rhombohedral-shaped Ge crystal with dimensions of 50 mm x 3 mm x 2 mm. This geometry allows multiple reflections of radiation to take place along the surface of the rubber. Figure 1 shows a schematic diagram of the ATR attachment.[7]

1. BLOW, C. M. Rubber Technology and Manufacture, The Buttworth Group, London, 1974.
2. DAVIDSON, H. W., WIGGS, K. C., CHURCHOUSE, A. H., MAGGS, F. A. P., and BRADLEY, R. D. Manufactured Carbon, MacLaren, London, 1968.
3. CHEN, C. H., KOENIG, J. L., SHELTON, J. R., and COLLINS, E. A. *The Influence of Carbon Black on the Reversion Process in Sulfur-Accelerated Vulcanization of Natural Rubber.* Rubber Chem. and Technol., v. 55, 1982, p. 103.
4. CORISH, P. J., J. Appl. Polym. Sci., v. 4, 1960, p. 86.
5. DEVLIN, E. F. *The Fourier Transform Infrared Spectrum of Cured, Black-Reinforced SBR.* J. Polym. Sci., Poly. Lett. Ed., v. 19, April 1981, p. 189.
6. HART, W. W., PAINTER, P. C., KOENIG, J. L., and COLEMAN, M. M., Appl. Spectrosc., v. 31, 1977, p. 220.
7. HARRICK, N. J. Internal Reflection Spectroscopy, Wiley-Interscience, New York, 1967.



$$d_p = \frac{\lambda/n_{Ge}}{2\pi[\sin^2\theta - (n_{SAM}/n_{Ge})^2]^{1/2}}$$

Figure 1. Schematic outline of the internal reflectance accessory.

d_p = depth of penetration into sample ($n_{Ge} > n_{SAM}$)
λ = wavelength of radiation in μm
n_{Ge} = refractive index of germanium
n_{SAM} = refractive index of sample
θ = angle of incidence of radiation

The IRE had an incident face angle of 60°. The depth of penetration varies as a function of frequency from approximately 1 μm at 4000 cm^{-1} to 1.2 μm at 400 cm^{-1}. Figure 2 shows a comparison of the depth of penetration for a germanium crystal and that of another often used internal reflectance crystal, KRS-5 (thallium bromide-iodide). This figure clearly shows the small penetration depth due to the use of the germanium crystal. Therefore, more IR radiation is permitted to reach the detector which was a severe problem previously encountered by other authors.[4,6] By co-adding a large number of spectral scans and utilizing a scale expansion of the resultant IR spectrum, all spectral features of the heavily loaded carbon black filled rubber can be identified.

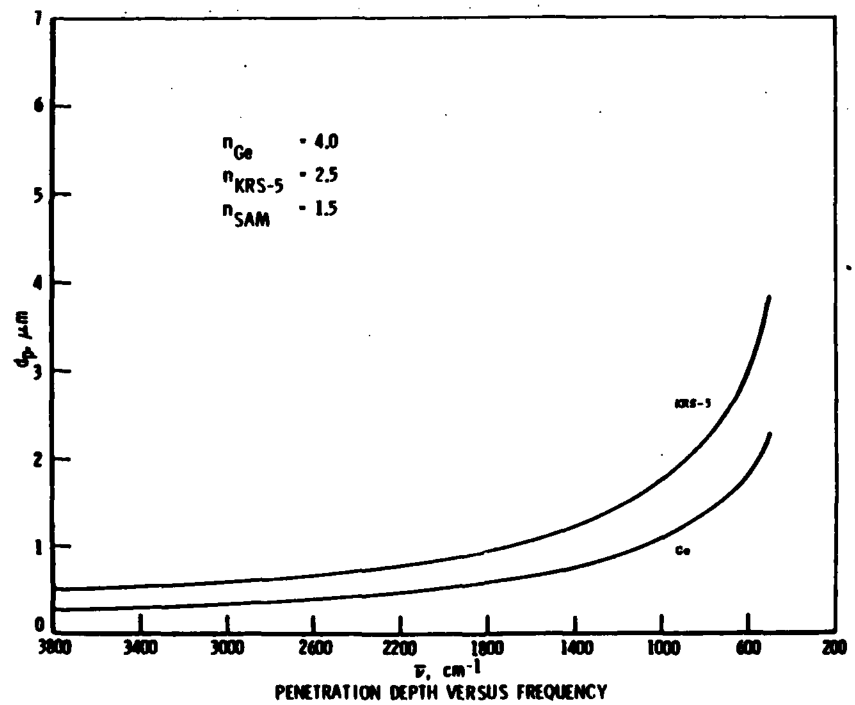


Figure 2. Depth of penetration profile of the infrared beam into the sample as a function of wave number.

All infrared spectra were measured on a Digilab FTS-10M Fourier transform infrared spectrometer. Spectra were recorded using 2048 co-added interferometric scans with a spectral resolution of 4 cm^{-1}.

Cured carbon black filled SBR samples were formulated by various rubber manufacturers and known to contain the same base SBR, containing approximately 24% bound styrene and processed with 50 per hundred carbon black. Samples were provided as bulk tensile sheets and cut to size. Sample formulations are given in Table 1.

Samples were prepared for IR analysis by soaking in acetone for one hour followed by soaking in methylene chloride for one hour. The samples were then allowed to dry in a vacuum oven at 100°C for one hour. This method of extraction serves to cleanse the elastomer surface of all additives and compounding ingredients while allowing the structural integrity of the elastomer to remain intact.

Table 1. SBR RUBBER FORMULATIONS IN PHR

	Control 22	Control	UOP 88	3 PTS UOP 88	Stereon 750
Stereon 750	-	-	-	-	100
SBR-1500	100	100	100	100	
Zinc Oxide	3	3	3	3	3
Stearic Acid	2	2	2	2	2
Phenyl-b-napthylamine	1	1	1	1	1
AgeRite Resin D	1	1	1	1	1
Carbon Black, HAF	50	50	50	50	-
N-cyclohexyl-2-benzothiayl Sulfonamide	1	1	1	1	-
Sulphur	1.75	1.75	1.75	1.75	2
UOP 88	-	-	coated	3	-
Aromatic Oil	-	-	-	-	37.5

RESULTS AND DISCUSSION

ATR is a complex technique governed by many fundamental optical laws. Some key factors for obtaining quality ATR spectra are as follows:

1. The quality of the internal reflectance spectrum depends on the nature of the contact between the IRE and the sample. It is desirable to distribute the pressure uniformly along the surface of the IRE. This is accomplished by use of a torque wrench which allows reproducible and uniform pressure of the sample onto the IRE.

2. Approximate matching refractive indices of the sample and the IRE are required if good spectral contrast is to be obtained. In our case, the refractive index of carbon black filled rubber has not been determined. One can assume that this number is quite high. Thus, a germanium IRE with a refractive index of 4.0 would be a more appropriate IRE than a crystal with a refractive index of 2.5 (KRS-5).

3. Accurate knowledge of a good reference spectra. In double-beam grating instruments the sample spectrum is ratioed against air. In FT-IR systems the reference spectrum is a digitally stored spectrum of a free standing IRE, which allows a fully compensated IR spectrum to be obtained.

Spectra of Heavily Loaded Carbon Black Filled SBR

Figure 3 shows a spectrum of a heavily loaded carbon black filled rubber before and after baseline correction. The sloping baseline in the uncorrected spectrum is due to the wavelength dependence of the depth of penetration of the infrared beam into the sample. This can be easily compensated by using a baseline correction program available in the operating software. Due to the limited amount of radiation reaching the detector and the strong absorption of the carbon black throughout the infrared region, a scale expansion was necessary to detect the infrared bands of the SBR. The result of this operation is seen in the bottom spectrum in Figure 3.

Figure 4 shows a comparison between the corrected carbon black filled SBR spectrum and that of a nonfilled SBR. The absorption bands in the spectrum of the filled SBR can clearly be identified with that of the unfilled sample spectrum. Absorptions due to the butadiene portion (967 cm^{-1}, 910 cm^{-1}) of the copolymer can be seen, as can the absorptions due to the styrene portion (698 cm^{-1}, 1498 cm^{-1}, 1601 cm^{-1}). Clearly, absolute identification of the base elastomer can be made from the IR spectrum obtained by this technique. The band located at 1539 cm^{-1} has already been assigned to the presence of zinc stearate,[8] which is formed during the curing process as a result of reaction between zinc oxide and stearic acid.

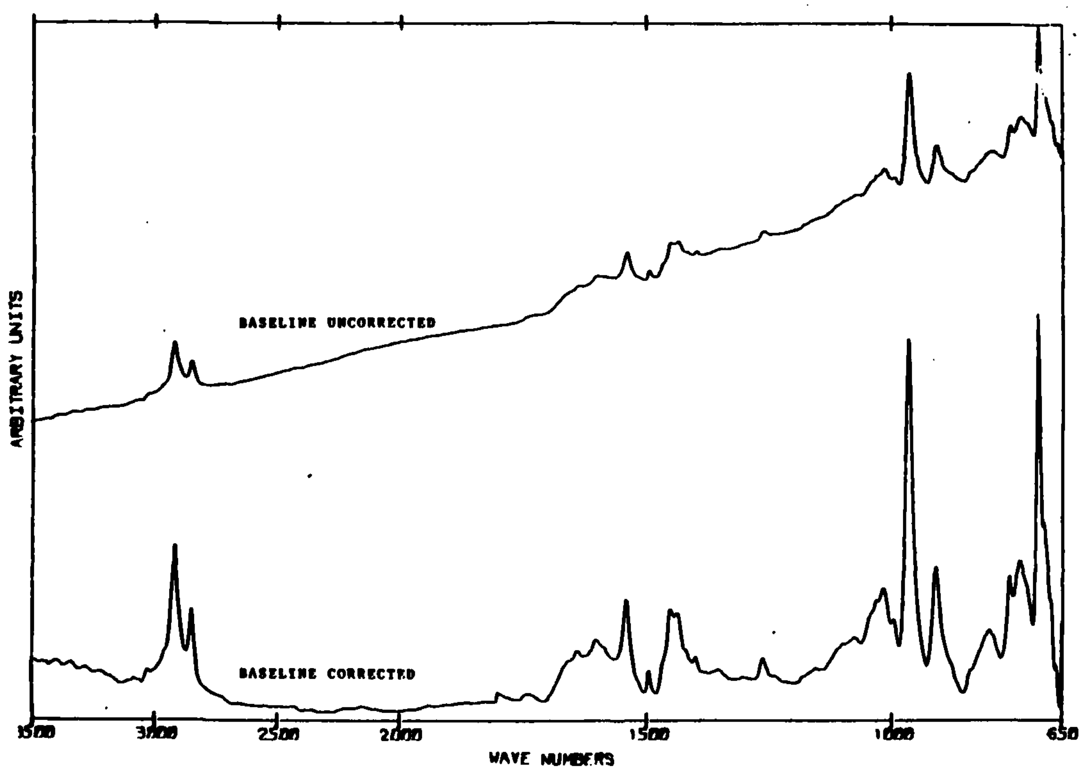


Figure 3. Top: uncorrected IR spectrum of carbon black filled SBR.
Bottom: baseline corrected IR spectrum of same SBR.

8. ANDRIES, J. C., ROSS, D. B., and DIEM, H. E. *Ozone Attack and Antiozonant Protection of Vulcanized Natural Rubber. A Surface Study by Attenuated Total Reflectance Spectroscopy.* Rubber Chem. Technol., v. 48, 1975, p. 41.

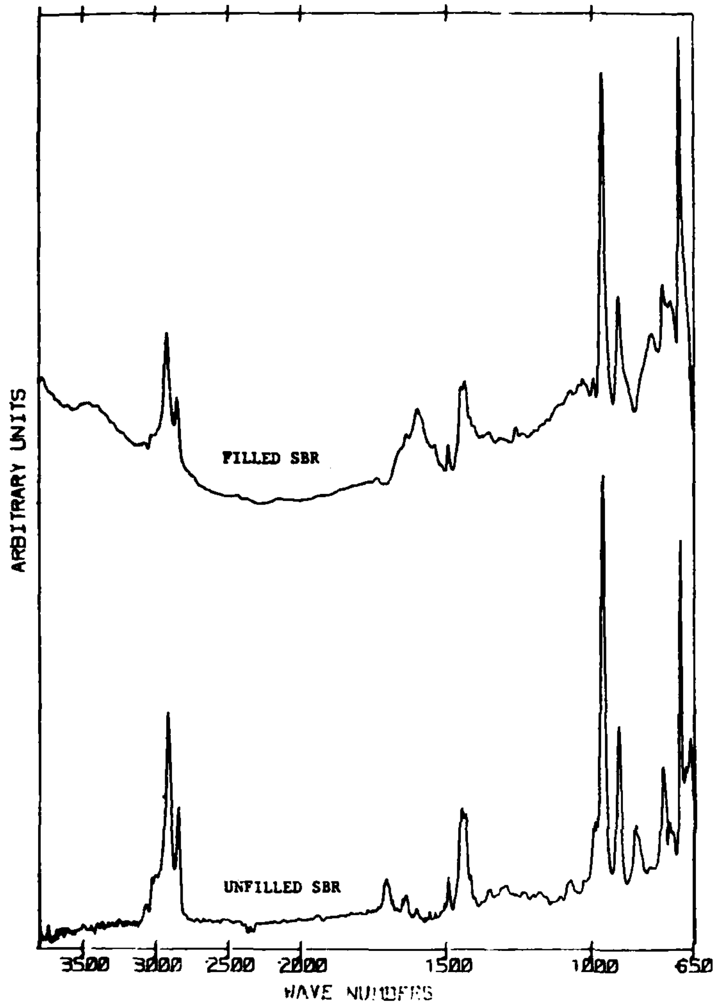


Figure 4. Comparison of two IR spectra. Top: carbon black filled SBR. Bottom: unfilled latex SBR.

Interaction Between Carbon Black and SBR

Figure 5 shows spectra of two different rubber formulations utilizing the same base SBR. The bottom spectrum is that of the unfilled SBR shown here for comparison. The top two spectra are spectroscopically identical as would be expected since the only differences between the two formulations are in the organic additives, which have been extracted out. This is an indication of the reproducibility that exists between different rubber formulations employing the same base rubber. Of particular interest in Figure 5 are the distortions of the various C-H bands along the polymer backbone. Absorbances at 698 cm^{-1} and 3065 cm^{-1}, attributed to various aromatic C-H motions, show changes in both their frequency maxima and their relative intensities. Similarly, absorbances at 2953 cm^{-1}, 2890 cm^{-1}, and 967 cm^{-1} attributed to the various C-H motions of the butadiene portion of the copolymer show intensity and positional shifts. These effects are thought to be due to the interaction between carbon black and the polymer. Although the exact nature of this interaction is beyond the scope of this report, it seems apparent that the polymer surface is strongly absorbed onto the carbon black.

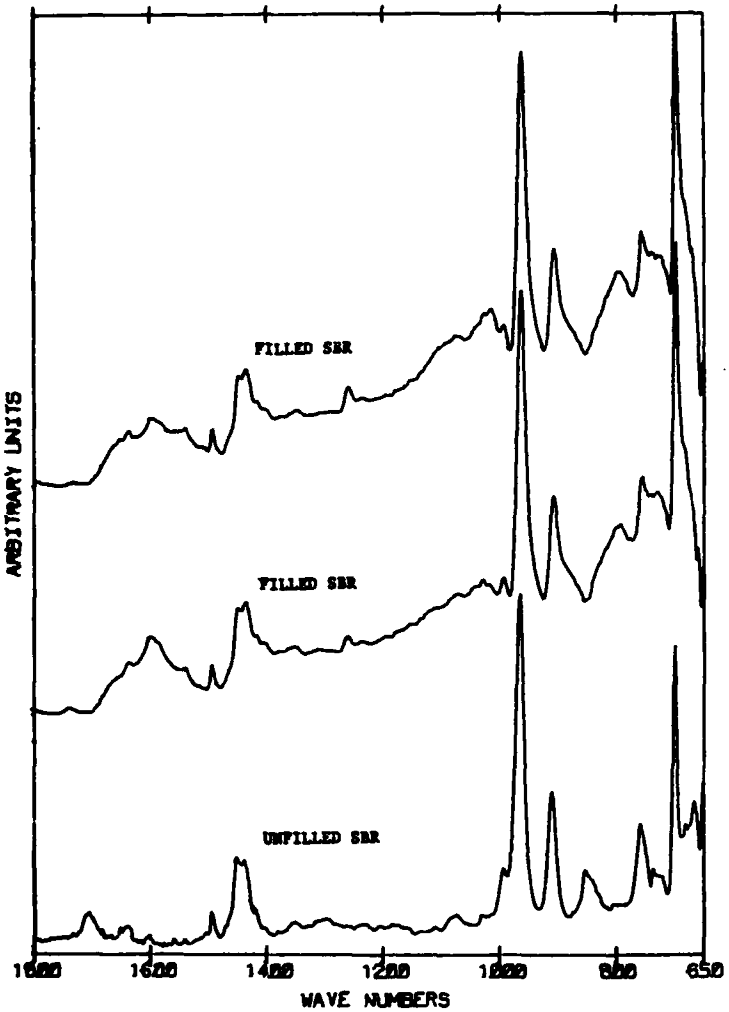


Figure 5. Spectral comparison between two differently formulated carbon black filled SBR and the raw unfilled SBR.

Figure 6 shows the expanded infrared region between 1350 cm^{-1} and 1480 cm^{-1} for the filled and unfilled elastomer. Clearly, the intensity of the 1433 cm^{-1} band increases with respect to the 1450 cm^{-1} band. These bands have previously been assigned to the CH_2 bending vibrations of the gauche and trans conformations, respectively,[9] about the C-C bond. It is interesting to note that only one band is modified by the addition of carbon black, indicating a preferred loss of conformation from the trans conformer to the gauche conformer.

Quantitative Determination of Bound Styrene in SBR

The spectra obtained using this sampling technique are of sufficient enough quality to allow determination of the percent bound styrene in the copolymer, SBR. The method of determination is to use calibrated IR band intensity ratios for unknown samples and compare them to the IR band intensity ratios for a series of standards. In this case, the 698 cm^{-1} band of styrene was ratioed against the 967 cm^{-1} band of butadiene. A calibration graph was constructed for three known standards. Such a graph is shown in Figure 7.

9. CRAMPELLI, F., and MANOVICHI, I. Gazz. Chim. Ital., v. 91, 1961, p. 1045.

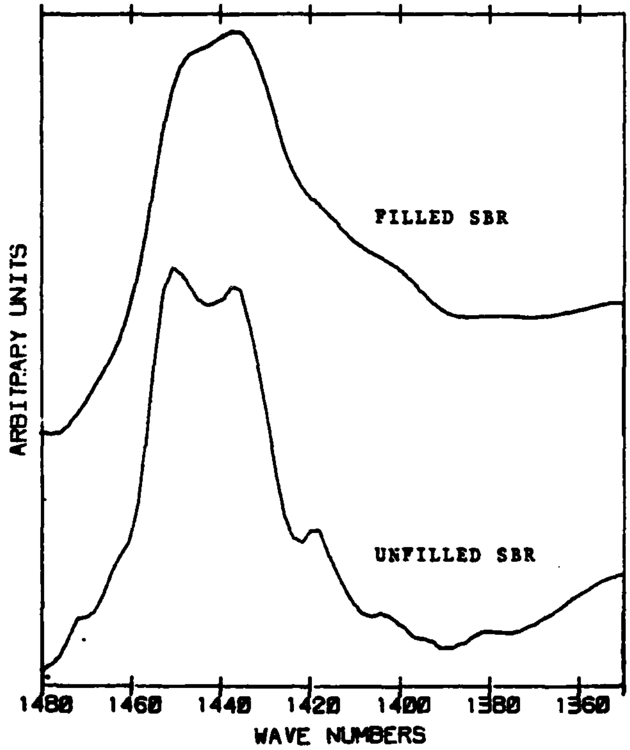


Figure 6. 1480 cm^{-1} - 1350 cm^{-1} region showing changes in band intensities upon addition of carbon black.

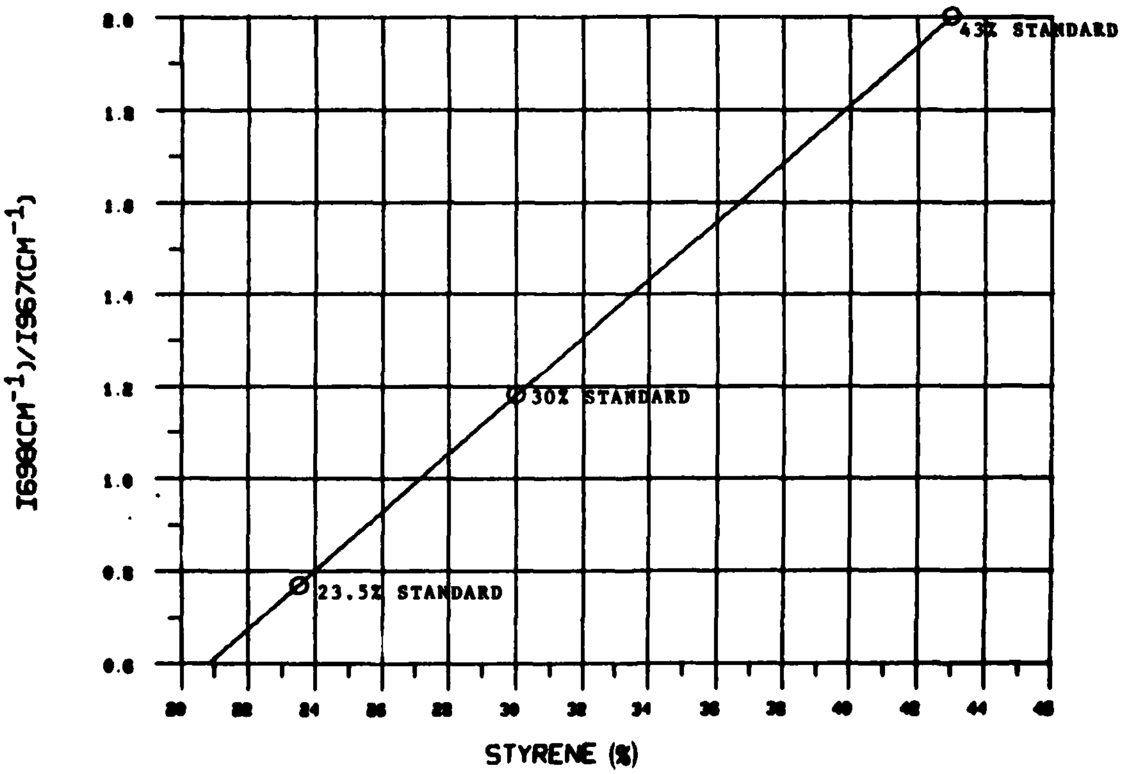


Figure 7. Typical calibration curve of IR band intensity ratios of 967 cm^{-1}/698 cm^{-1} versus percent-bound styrene in SBR.

Table 2 summarizes the results of a series of known cured carbon black filled samples analyzed in this way. As can be seen by this table, excellent agreement is shown between the known amount of styrene and the determined amount of styrene. These values show a maximum variation of between ±2.5%.

Table 2. QUANTITATIVE ANALYSIS OF STYRENE-BUTADIENE COPOLYMERS

Sample	Known Styrene (%)	Determined Styrene (This Work) (%)
Control 22	23.5	24.7
UOP 88	23.5	24.5
3 PTS UOP 88	23.5	23.5
Control	23.5	24.5
TACOM L	23.5	22.8
Stereon 750	18.0	20.2

Identification of Antioxidants

For a complete analysis of a formulated elastomer system, it is useful to identify the antioxidant that has been used in processing. The small amount of antioxidant present (typically up to 3% by weight) in the formulation makes it necessary to extract the antioxidant as performed in the experimental section and concentrate it down. This extract contains various amounts of processing components, but is primarily composed of the antioxidant.

The solvent can be evaporated onto a KBr salt plate and an infrared spectrum taken. Figure 8 shows an example of the spectrum one obtains from this extracted residue. This material has been identified as an N,N° substituted phenylene diamine antioxidant commonly used in elastomer formulations.

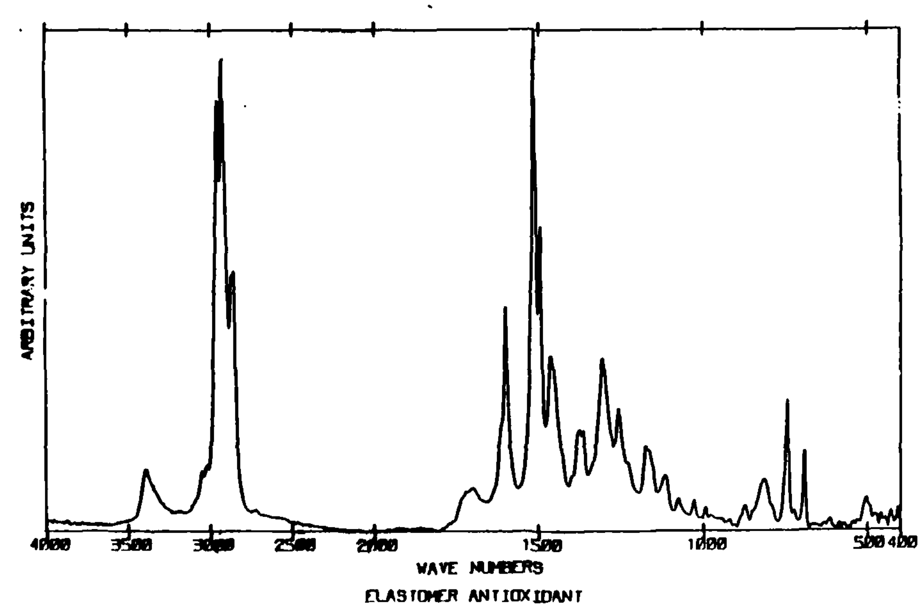


Figure 8. IR spectrum of rubber extract.

CONCLUSION

It has been shown that quality infrared spectra of heavily loaded (50 per hundred) carbon black filled styrene-butadiene rubbers can be measured by the use of a standard ATR sampling accessory. The utilization of Ge as the internal reflectance element allows more radiation to reach the detector. The resultant spectra yields absolute identification of an unknown elastomer.

The spectra obtained are of sufficient quality to allow subtle spectral changes of the elastomer to be detected. These changes are associated with the interaction of the polymer with the carbon black filler.

Additionally, FT-IR provides the advantage of a rapid and easy method for identification of quantitative differences in composition between standard and unknown styrene-butadiene rubbers by absorption band ratios. This has been shown to be accurate to ±2.5% bound styrene on samples tested.

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Infrared spectra have been obtained of a heavily loaded (50 per hundred) carbon black filled styrene-butadiene rubber using a Fourier transform infrared spectrometer. Attenuated total reflectance (ATR) spectroscopy sampling employing a germanium (Ge) internal reflectance element was found to yield quality infrared spectra where other techniques have failed. Subtle changes were observed between the spectra of the filled and unfilled styrene-butadiene rubber (SBR). These changes are attributed to the interaction between polymer and filler.

Army Materials and Mechanics Research Center,
Watertown, Massachusetts 02172-0001
DIRECT ANALYSIS OF HEAVILY LOADED CARBON BLACK
FILLED STYRENE-BUTADIENE RUBBER USED IN TANK TRACK
PADS BY FT-IR -
James M. Sloan and Joseph P. Clements
Technical Report AMMRC TR 84-25, June 1984, 11 pp -
illus-tables, D/A Project 1T161101A91A

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