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APPENDICES

APPENDIX A

Determination of Ash Content

Determination of ash is followed by RRIM B6

A-1 Explanatory Notes

The ash from NR contains in varying proportions the oxides, carbonates and phosphates of potassium, magnesium, calcium, sodium and other trace elements. The ash may also contain silica or silicates arising either from the rubber or form extraneous foreign matter to an extent depending on the history of the material. The specified ash content represents a minimal figure for the amount of mineral matter present in the rubber. The method of determination involves wrapping the test portion in filter paper and incinerating in a muffle furnace at about 550°C.

A-2 Apparatus and Material

(a) Crucible, of nominal capacity 50 ml.
(b) Muffle furnace, with pyrometer and thermostatic control
(c) Ashless filter paper of 15 cm. Diameter
(d) Vacuum desiccator of 20 cm. Or 25 cm. Diameter
(e) Tong for handling crucible

A-3 Procedure

Weigh, to the nearest 0.1 mg, a 5 g to 10 g portion of the homogenised rubber. Wrap in ashless filter paper and place in a crucible, which has been previously ignited and weighed. Introduce the crucible into muffle furnace controlled at a temperature of 550°C ±20°C until free form carbon (approximately 2 h to 4 h) When ashing is complete, allow the crucible to cool in a desiccator and then weigh to the nearest 0.1 mg.

A-4 Calculation

Calculate as follows:

$$\text{Ash (\%)} = [(A-B-C)/D] \times 100$$

Where A is the weight of crucible plus ash;

B is the weight of empty crucible;

C is the weight of ash from filter paper;

D is the weight of sample taken.

APPENDIX B

Nitrogen content data and statistical deviation

Table B.1 Nitrogen content of saponified rubber with 5% w/v NaOH in the presence of 0.1 and 0.2 %(w/v) Triton X-100 at 70°C for 3 h. followed by single or double centrifugation.

Triton X-100, % (w/v)	No. of centrifugation	Nitrogen content, %				
		Exp. 1	Exp. 2	Exp. 3	Avg.	SD
0.1	1	0.025	0.024	0.028	0.0067	0.002
0.1	2	0.0055	0.0058	0.0060	0.0210	0.002
0.2	1	0.021	0.019	0.023	0.0058	0.00025
0.2	2	0.0034	0.0035	0.0038	0.0036	0.00021

Table B.2 Nitrogen content of DPNR by saponification with NaOH/Triton X-100

Triton X-100, %(w/v)	NaOH/Triton X-100	Nitrogen content, %				
		Exp. 1	Exp. 2	Exp. 3	Avg.	SD
0.1	Triton X-100	0.015	0.018	0.018	0.017	0.0017
0.1	NaOH and Triton X-100	0.0055	0.0058	0.006	0.0058	0.0002
0.2	Triton X-100	0.011	0.013	0.011	0.012	0.0011
0.2	NaOH and Triton X-100	0.0034	0.0035	0.0038	0.0036	0.0002

Table B.3 Nitrogen content of saponified rubber with 1-5% (w/v) NaOH

Exp.	NaOH, % (w/v)	Nitrogen content, %				
		Exp. 1	Exp. 2	Exp. 3	Avg.	SD
Control	0	0.544	0.541	0.538	0.541	0.003
1	1	0.0354	0.0341	0.0355	0.035	0.00078
2	2	0.031	0.0287	0.0282	0.0293	0.00149
3	3	0.0251	0.0235	0.0228	0.0238	0.00118
4	4	0.016	0.0195	0.0188	0.0181	0.00185
5	5	0.0177	0.0164	0.0169	0.017	0.00066

Table B.4 Nitrogen content of saponified rubber observed by different surfactant concentration and step of surfactant adding

Concentration of Triton x-100,%(w/v)			Nitrogen content, %				
Saponification (1)	Washing step		Exp1	Exp2	Exp3	Avg.	SD.
	(2)	(3)					
0.1	0	-	0.0055	0.0058	0.006	0.0058	0.00025
0.1	0	-	0.01	0.0097	0.0097	0.0098	0.00017
0	0.1	-	0.0207	0.0202	0.0211	0.0207	0.00045
0	0.1	-	0.048	0.045	0.046	0.0463	0.00152
0.2	0	0.2	0.0034	0.0035	0.0038	0.0036	0.00020
0.2	0	0	0.007	0.0063	0.0068	0.0067	0.00036
0	0.2	-	0.012	0.011	0.0142	0.0124	0.0016
0	0.2	-	0.0.12	0.011	0.0142	0.0126	0.0023
0.4	0	0.4	0.00014	0.0002	0.00021	0.0002	0.00006
0.4	0	0	0.0032	0.004	0.0032	0.0038	0.00046
0	0.4	0.4	0.0068	0.008	0.0077	0.0075	0.00062
0	0.4	0	0.0066	0.0081	0.0072	0.0073	0.00075

APPENDIX C

Intensity ratio of ^{13}C-NMR signals of fresh and saponified rubber and calculation

Table C.1 Intensity ratio of ^{13}C-NMR signals at 24 and 29.71 ppm of saponified rubber with 1-5 %(w/v) NaOH

Experiment	NaOH, %(w/v)	Weight of peak			
		W_{24}	$W_{real\ 24}$	W_{29}	$W_{real\ 29}$
Control field rubber	0	0.0035	2.270	0.1349	0.0899
1	1	0.0975	1.625	0.1240	0.0827
2	2	0.0699	2.330	0.1048	0.0699
3	3	0.0828	1.656	0.1305	0.0870
4	5	0.0825	2.750	0.1982	0.1321

Where; $W_{real\ 24} = W_{24}$*(Y-gain/X-expanded)

$W_{real\ 29} = W_{29}$*(Y-gain/X-expanded)

APPENDIX D

Ester content calibration curve and calculation of ester content of saponified rubber from calibration curve

Table D.1 Peak height ratio of standard ester content for calibration curve

Ester content (mmol/kg rubber)	Peak height ratio
3.28	0.0251
9.26	0.1230
9.81	0.0901
14.99	0.9450
28.71	0.2080
40.16	0.2730

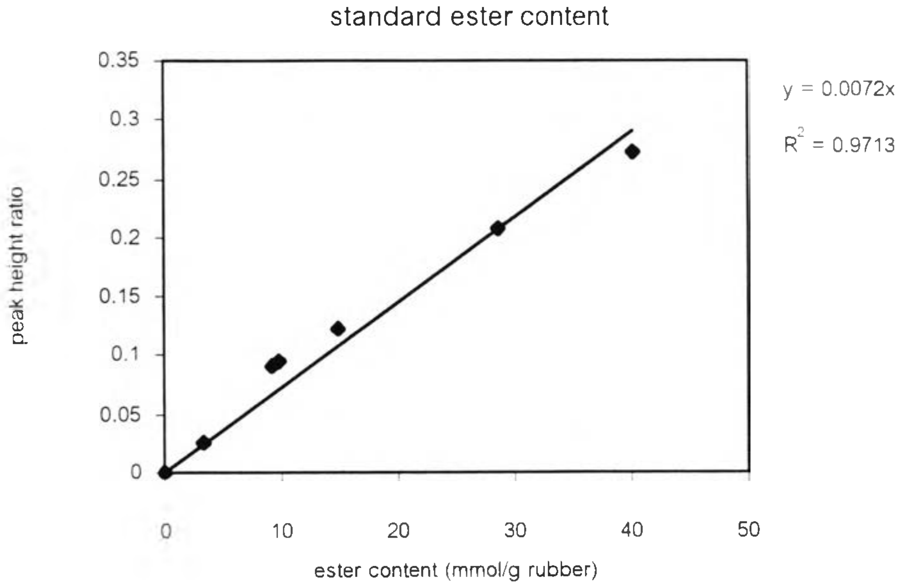

Figure D.1 Standard ester content calibration curve.

Table D.2 Peak height ratio of saponified rubber

Experiment	NaOH, %(w/v)	Peak height (1739 cm^{-1})	Peak height (1664 cm^{-1})
Control field rubber	0	0.199	2.459
1	1	0.096	1.105
2	2	0.124	0.925
3	3	0.162	1.123
4	4	0.089	0.570
5	5	0.380	2.047

VITA

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