

Infrared Analysis of o-Tricresyl Phosphate in Paraffinic Mineral Oil

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CS-95

Component			Range %	Accuracy %	λ or ν B. L. Pts.	Slit (mm) $\Delta\lambda$ or $\Delta\nu$	Concn. mg/ml Length mm
No.	Name	Formula					
1	o-Tricresyl phosphate	$C_{21}H_{21}PO_4$	0-4	± 0.05	0.11 9.5- 11.1	0.11 0.058 μ	100 0.1

Instrument: Perkin-Elmer Model 21, NaCl prism
Sample Phase: Full strength

Cell Windows: NaCl

Absorbance Measurement: Base line ☒ Point ☐

Calculation: Inverse matrix ☐ Successive approx. ☐
Graphical ☒

Relative Absorbances^a-Analytical Matrix:

Component/ λ	
1	10.35 μ 24.6

Material Purity: Reference compound 99+ % pure

Comments: Some oils have a small background absorption in the region of 10.3 μ . This may be due to the natural absorption of the oil or to other additives used to give the oil special properties. Absorptive interferences are usually small and corrections easily made by standard techniques.

^a Relative absorbances are given as the slope of the Beer's law concentration curves used expressed in terms of absorbance per 100% of constituent.

Determination of 1,2-Ethanediol, 1,2-Propanediol, and 2,2'-Dihydroxyethyl Ether

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Component			Range %	Accuracy %	λ or ν B. L. Pts.	Slit (mm) $\Delta\lambda$ or $\Delta\nu$	Concn. mg/ml Length mm
No.	Name	Formula					
1	1,2-Ethanediol	$C_2H_6O_2$	0-100	± 2	863 cm^{-1}	0.70 0.122 μ	20 0.5
2	1,2-Propanediol	$C_3H_8O_2$	0-100	± 2	837	0.70 0.120 μ	20 0.5
3	2,2'-Dihydroxyethyl ether	$C_4H_{10}O_2$	0-100	± 2	1131	0.45 0.151 μ	20 0.2

Instrument: Perkin-Elmer Model 12 C, NaCl prism
Sample Phase: Solution in acetonitrile

Cell Windows: NaCl

Absorbance Measurement: Base line ☐ Point ☐

Calculation: Inverse matrix ☒ Successive approx. ☐
Graphical ☐

Relative Absorbances^a-Analytical Matrix:

Component/ λ	863 cm^{-1}	837 cm^{-1}	1131 cm^{-1}
1	35.3	7.4	1.95
2	4.6	22.9	11.4
3	9.5	4.9	28.1

Material Purity: Reference compounds were rectified commercial products

^a Relative absorbances are given as the absorbance which a 1 gram per ml. solution would have in the cell in which the measurements are made.

Acrylonitrile in Acrylonitrile-Butadiene Copolymer

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Component			Range %	Accuracy %	λ or ν B. L. Pts.	Slit (mm) $\Delta\lambda$ or $\Delta\nu$	Concn. mg/ml Length mm
No.	Name	Formula					
1	Acrylonitrile (bound)	$-C_3H_3N-$	20-40	0.6	2920 cm^{-1} 2400 2230 2200	0.0520 30 cm^{-1} 0.0060 4.0 cm^{-1}	

Instrument: Perkin-Elmer Model 21, NaCl prism
Sample Phase: Solid film, hot-pressed

Cell Windows: Mica sheets

Absorbance Measurement: Base line ☒ Point ☐

Calculation: Inverse matrix ☐ Successive approx. ☐
Graphical ☒

Relative Absorbances^a-Analytical Matrix:

Component/ λ

$[A_{2920}/A_{2230}]^{1/2}$ plotted against A_{2230} gives a family of curves for varying per cent acrylonitrile. For the range 20-40%, abscissa range is 0.30-0.60, ordinate: 0.930-1.422

Material Purity: Purified polymers, analyzed by Dumas method for nitrogen, used as standards. Calibration accuracy estimated $\pm 0.5\%$.

Comments: Singlicate precision $\sigma = 0.2\%$. Soap and antioxidant less on accuracy.

^a Relative absorbances are given as the slope of the Beer's law concentration curves used expressed in terms of absorbance per 100% of constituent.

cis-1,4-Isomer in 1,4-Polyisoprene

R. M. B. SMALL, Polymer Corp., Ltd., Sarnia, Ontario, Canada

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Component			Range %	Accuracy %	λ or ν B. L. Pts.	Slit (mm) $\Delta\lambda$ or $\Delta\nu$	Concn. w/w % Length cm
No.	Name	Formula					
1	cis-1,4-Polyisoprene	$-C_5H_8-$	0-100	5	2.46 μ	0.096	2.5 1.0

Instrument: Beckman Model DK 2

Sample Phase: Solution in carbon tetrachloride

Cell Windows: Corex

Absorbance Measurement: Base line ☒ Point ☐

Calculation: Inverse matrix ☒ Successive approx. ☐
Graphical ☐

Relative Absorbances^a-Analytical Matrix:

Component/ λ	2.46 μ (conc. w/w. % at 2.5%)
cis-1,4-Polyisoprene	0.166
trans-1,4-Polyisoprene	0.127

Note: Sample run differentially against a 2.5% solution of purified Hevea.

Material Purity: Extensively purified Hevea and Balata assumed 98% cis- and 98% trans-1,4-polyisoprene

Comments: Insensitivity of method to microstructure changes relative to impurity effects makes purification and thorough drying of sample essential. Light scattering creates sloping background. Value at 2.46 μ calculated by extrapolating a plot of absorbance vs. λ^{-4} through the points $\lambda = 1.000, 1.100, 1.325, 1.550\lambda$.

Spectral slit width not available.

^a Relative absorbances are given as the slope of the Beer's law concentration curves used expressed in terms of absorbance per 100% of constituent.

