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CHARACTERIZATION AND IDENTIFICATION OF NITROGEN-CONTAINING CHEMICAL FIBRES AND WOOL BY INFRARED SPECTROMETRIC METHOD *

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DUDE JA VILLA ISELOOMUSTAMINE NING IDENTIFITSEERIMNE INFRAPUNASE
SPEKTROSKOOPIA MEETODIL

О. КИРРЕТ, Лилья ЛАХЕ, Эне КИРЬЯНЕН. ХАРАКТЕРИСТИКА И ИДЕНТИФИКАЦИЯ СИНТЕ-
ТИЧЕСКИХ АЗОТСОДЕРЖАЩИХ И ШЕРСТЯНЫХ ВОЛОКОН МЕТОДОМ ИК-СПЕКТРО-
СКОПИИ

Introduction

Data are presented on possibilities of identifying wool and N-containing synthetic fibres by the infrared spectroscopic method.

The following commercial fibres were studied: aromatic and alicyclic polyamides Nomex and Qiana, polyurethane Fiz-Elastomer, polyacrylonitrile fibres and 10 samples of wool from different regions of the USSR. Besides using infrared spectra and computer, an elemental analysis of all fibre samples was performed, which provided valuable additional information. The characteristic data on the absorption bands of the fibres are in fair accordance with the results reported by D. O. Hummel [1].

Experimental and results

Infrared spectra were taken on a spectrophotometer «Specord IR 75» (Zeiss, GDR) in the region of 400—4000 cm^{-1} using the KBr pellet method. The fibres were characterized on the basis of the absorption bands of their infrared spectra; of them, only most characteristic and highly intense ones and those of medium intensity were chosen (Table 1). In addition to the absorption bands of fibres, the relative intensities are given.

Identification is based on the comparison of wave numbers and weights (intensities) of the bands. The wave numbers compared are considered different if the difference exceeds ± 5 cm^{-1}. The infrared spectra measured by the authors are given in Fig. 1 (for corresponding wave numbers see Table 1 in [2, 3]).

Table 2 shows that pure polyacrylonitrile fibre has no bands in the region of carbonyl groups. In the case of modified polyacrylonitrile there is a very strong absorption band in the region of 1722—1735 cm^{-1}. In literature, the absorption bands 1729 cm^{-1} (average), 1362 and 1228 cm^{-1} (average) have been correlated with the acetate structure, correspondingly with the C=0 stretching vibration.

Difficulties arise when, besides vinyl acetate, also metacrylic acid and its derivatives are used as comonomers whose molecules contain carboxylic, —CH$_3$- and —COC-groups, and the absorption bands of their

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Table 1

Characteristic absorption bands of some N-containing fibres, cm⁻¹

Qiana	Nomex	Fiz-Elastomer	Wool	Polyacrylo-nitrile
	570 W		550—650 S	
	680 M			
	720 W			
	780 W	760 W		
	855 W	800 W		
900 W	1075 M	1020 W	1035 M	1015 W
950 W		1040 W		
1005 W	1230 VS	1080 W	1075 M	1080 M
1030 M	1300 S	1140 M	1115 W	
1130 M	1320 S	1180 M	1170 W	1180 M
1220 M		1230 M	1235 M	1250 M
	1405 VS	1320 M		
1260 M		1380 M	1390 M	1382 M
1360 M	1470 VS	1420 W		1455 VS
1450 S	1520 VS	1460 M	1450 S	
1535 VS	1605 VS	1535 S	1520—30 S	
		1605 M		
1650 VS	1650 VS	1675 M	1635—45 S	
		1730 S		1735 VS
				2245 VS
2855 VS	2860 W	2865 W	2860 M	2863 M
2925 VS	2935 W	2930 M	2930 S	2915 VS
	2960 W	2955 M	2960 M	2960 SH
3080 M	3065 W		3070 M	
3300—15 VS	3300—15 W	3300—15 W	3280 S	
3445 S	3420 M	3425 W	3300—3400 S	

Abbreviations: VS — very strong, SH — shoulder, S — strong, M — middle, W — weak.

Table 2

Characteristic bands of pure polyacrylonitrile [1]

2950 VS		1355	
2930 SH		1247 M	
2870 M		1073 S	
2237 VS		778 M	
1447 VS		537 M	
1362 M			

infrared spectra coincide with those of the acetate structure. A differentiation between the above structures by conventional infrared measurements is practically impossible, suggesting the need for some other method.

From Table 3 it is evident that by elemental analysis on a «Hewlett-Packard 185» analyzer, the sum of C, H and N does not amount to 100 %, the difference being caused by oxygen, sulfur and chlorine contained in comonomers, and by flatting agents. As comonomers, various chemical

Table 3

Elemental analysis of some investigated fibres *

| Trade name	Provenance	Elemental composition, %			
		C	H	N	C+H+N*
Fiz-Elastomer	FRG	52.98	6.26	3.26	62.50
Nitron	USSR	67.61	5.69	25.36	98.66
Prelana	GDR	67.06	5.77	25.89	98.72
Nomex	USA	69.24	4.18	11.29	84.71
Qiana	USA	72.66	10.67	6.59	89.92
Wool	USSR	48.32	7.07	15.89	71.26

* The rest — O.
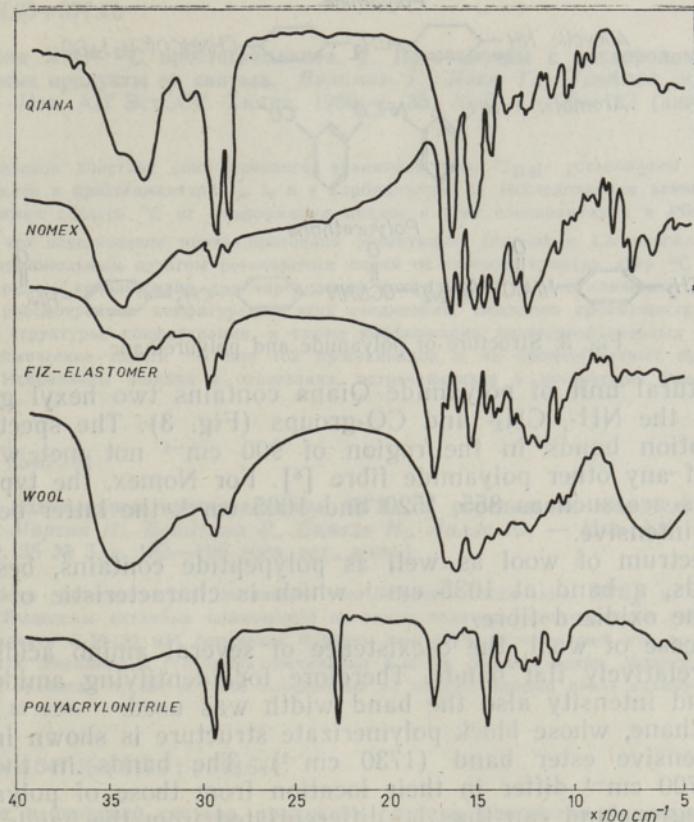
Fig. 1. Infrared spectra of some N-containing fibres.

compounds are used, such as vinyl pyrrolidone, alkyl vinyl sulfone, styrene sulfonic acid, vinyl phosphoric acid, polymerized dicarboxylic acid, etc.

To establish a calibration curve for the nitrile $(C≡N)$-group concentration versus the 2240 cm^{-1} absorbance band, several standard pellets were prepared by mixing known amounts of acrylic fibre and wool (Fig. 2) using the 13 mm KBr pellet technique [4—6]. The absorption intensities were calculated by a base-line method according to the method of Friedel [7].
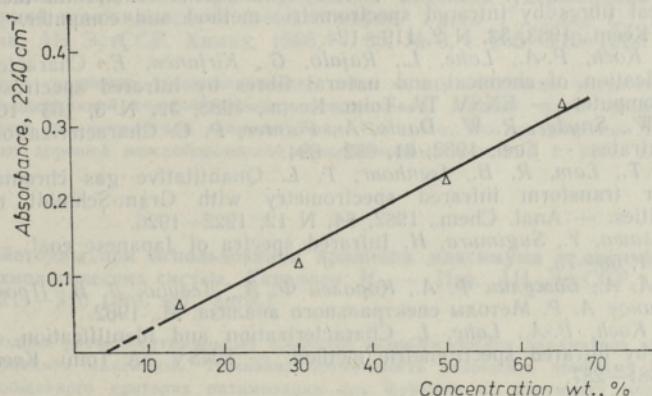

Fig. 2. Content of nitrile group in fibre.

Polyamide

Alicyclic NH—⟨⟩—CH_2—⟨⟩—$CHNHCO(CH_2)_{10}CO$

Aromatic NH $NHCO$ CO

Polyurethane

CH_2—⟨⟩—$HNCO$—$(CH_2)_4$—$OCNHN$—⟨⟩—CH_2—⟨⟩—HNC
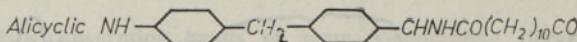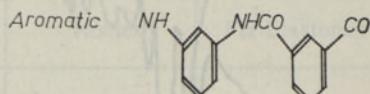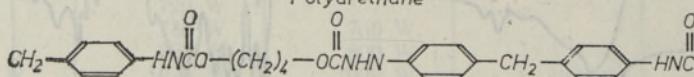
Fig. 3. Structure of polyamide and polyurethane.

A structural unit of polyamide Qiana contains two hexyl groups in addition to the NH-, CH_2- and CO-groups (Fig. 3). The spectrum has two absorption bands in the region of 900 cm^{-1} not met with in a spectrum of any other polyamide fibre [8]. For Nomex, the typical aromatic bands are such as 855, 1520 and 1605 cm^{-1}, the latter being very sharp and intensive.

The spectrum of wool as well as polypeptide contains, besides the amide bands, a band at 1035 cm^{-1} which is characteristic of the SO-group in the oxidized fibre.

In the case of wool, the coexistence of several amino acids results in broad, relatively flat bands. Therefore for identifying amido bands, besides band intensity also the band width was used.

Polyurethane, whose block polymerizate structure is shown in Fig. 3, has a intensive ester band (1730 cm^{-1}). The bands in the region of 1600—1700 cm^{-1} differ in their location from those of polyamide of the same region, and can thus be differentiated from the bands of polyamide and natural wool.

Conclusions

In conclusion it may be said that wool and the N-containing synthetic fibres with chemically similar structural groups can be characterized on the basis of their infrared spectra using characteristic bands of amide, aromatic and nitrile groups. More detailed identification of the modified polymers and copolymers is possible by using infrared spectra in combination with some other analytical methods.

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