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STEP

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F031/F004

AUTHOR: Chang, Te-ho (1728/1795/7901), Ch'in, Wen-mao (4440/2429/2021),
and Ch'ien, Jen-yüan (6929/0086/0337)

TITLE: Infrared spectroscopic investigation of the cis-trans isomerization
of the polyester from maleic anhydride-ethylene glycol

PERIODICAL: Hua Hsüeh Hsüeh Pao, v. 28, no. 3, 181-186

TEXT: Experiments were made by the authors to determine the relative contents of cis- and trans-forms in polymers by analysis and comparison of intensities of the respective infrared absorption bands. Diethylmaleate and diethyl fumarate were used as model compounds. The following experiments were made: 1. Preparation of cis-diethylmaleic acetate: React cis-maleic anhydride, anhydrous ethyl alcohol, benzene and concentrated sulphuric acid and separate out the phenyl layer, neutralize with sodium bicarbonate and dry by means of calcium sulphate. Fractionate at 219-220°C. 2. Preparation of trans-diethylmaleic acetate. Follow the same process but use cis-

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maleic anhydride as material. Fractionate at 213-215°C. 3. Preparation of poly-diethylmaleic acetate. Place 196 mol cis-maleic anhydride c. p. and 124 mol ethylene glycol c. p. in a flask for condensation polymerization at temperatures 100°C, 130°C, and 160°C under the protection of nitrogen. Take 15 ml of the reactant and determine the infrared absorption spectra: a. The product from 100°C reaction for 2 hrs shows entirely in the cis-form, b. The product from 160°C reaction for 30 hrs and from 200°C reaction for 10 hrs show entirely in the trans-form. 4. Determination of acidity values: Take 0.2-0.3 gm of polyester, dissolve it in 15 ml benzene-alcohol (1:1) mixture, and titrate by 0.2 N NaOH-alcohol solution to the end. 5. Drawing the infrared spectrograms. A Zeiss UR-10 infrared spectrophotometer was used. An NaCl prism was employed for the range of 700-800 cm^{-1} of the spectrobands and a lithium fluoride prism was used for those in the range of 2800-3100 cm^{-1} . A 0.4 mm thick liquid bath was used to draw cis- and trans-diethyl maleic acetates and a quantity of NaCl was placed in the bath to offset absorption of NaCl. 6. Determination of the viscosity of the polyester. This is done by using the Ferranti-Shirley conic plate

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at 40°C. The following results were obtained: 1. With the model compounds, the characteristic infrared bands of the cis- form polyester from maleic anhydride-ethylene glycol were identified as 825, 1410, 3052 cm^{-1} and of the trans- form, 780, 1371, 3072 cm^{-1} . 2. The ratio of the optical densities of the 825 and 780 cm^{-1} bands has been chosen for determination of the ratio of cis- and trans- forms present in the polyester. 3. Increased temperature and duration of polyesterification apparently increase the trans- form polyester. The cis- trans isomerization was shown to be a first order reaction with an activation energy of 19 KCal/mole. 4. The large increase of viscosities of the reacting mixture during polyesterification of maleic anhydride and ethylene glycol is mainly due to an increase of trans-content of the polyester formed. There are 6 figures and 1 table. References in English include: G. M. Kline, "High Polymers, vol. XIII, Pt. I. Analysis of monomers and polymeric materials" p. 36 Interscience, New York, 1959; W. L. Walton and R. B. Hughes, Ana. Chem. 28, 1388 (1956).

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