

UNCLASSIFIED

AD NUMBER
AD400767
NEW LIMITATION CHANGE
TO Approved for public release, distribution unlimited
FROM Distribution authorized to U.S. Gov't. agencies and their contractors; Foreign Government Information; 09 NOV 1961. Other requests shall be referred to US Library of Congress, Attn: Aerospace Technology Division, Washington, DC.
AUTHORITY
ATD ltr, 2 Dec 1965

THIS PAGE IS UNCLASSIFIED

UNCLASSIFIED

AD **400 767**

DEFENSE DOCUMENTATION CENTER

FOR

SCIENTIFIC AND TECHNICAL INFORMATION

CAMERON STATION, ALEXANDRIA, VIRGINIA



UNCLASSIFIED

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

400767

21

STEP

C/002/62/028/003/003/003
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. Diethylmelate 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-

Card 1/4

Infrared spectroscopic investigation ...

C/002/62/028/003/003/003
F031/F004

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

Card 2/4

Infrared spectroscopic investigation ...

C/002/62/028/003/003/003
F031/F004

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).

Card 3/4

Infrared spectroscopic investigation ...

C/002/62/028/003/003/003
F031/F004

ASSOCIATION: Chung Kuo K'o Hst'eh Y'uan Hua Hst'eh Yen Chiu So (Chemistry Institute, Chinese Academy of Sciences)

SUBMITTED: November 9, 1961

Card 4/4