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Publisher: Taylor & Francis

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## Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gmcl16>

## Synthesis of Poly (Vinylene Sulphide)

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Version of record first published: 17 Oct 2011.

To cite this article: Yukihiro Ikeda, Masaru Ozaki & Tatsumi Arakawa (1985): Synthesis of Poly (Vinylene Sulphide), *Molecular Crystals and Liquid Crystals*, 118:1, 431-434

To link to this article: <http://dx.doi.org/10.1080/00268948508076252>

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## SYNTHESIS OF POLY(VINYLENE SULPHIDE)

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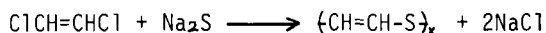
**Abstract** Pure and high molecular poly(vinylene sulphide) was synthesized from 1,2-dichloroethylene and sodium sulphide anhydride using dimethyl sulfoxide as a solvent.

### INTRODUCTION

Poly(vinylene sulphide),  $\{\text{CH}=\text{CH}-\text{S}\}_x$ , (PVS) is the simplest sulphur containing unsaturated polymer. We have reported a preparation method of this novel polymer<sup>1</sup>. We have recently succeeded to prepare higher and more pure PVS by using water-free materials and dimethyl sulfoxide(DMSO) as a solvent.

### PREPARATION OF PVS

PVS was prepared by the condensation polymerization of sodium sulphide anhydride and 1,2-dichloroethylene as follows,



Dimethyl sulfoxide(DMSO) was used as a solvent. DMSO(90ml) and  $\text{Na}_2\text{S}$ (15.2g) were added to a reactor equipped with a powerful stirrer under an argon atmosphere. The sodium sulphide solution was stirred vigorously at  $20\sim 50^\circ\text{C}$ , and then trans- or cis-1,2-dichloroethylene(19.3g) was added. After 12~15 hours for cis-1,2-dichloroethylene and 70~100 hours for trans-1,2-dichloroethylene, the reaction mixture was filtered and the filtrate poured into a large quantity of methanol. Precipitation of PVS is given in powder form which has light brown colour and it can be obtained as a film by heat pressing at above  $100^\circ\text{C}$ .

The structure of PVS was examined by  $^{13}\text{C}$ -n.m.r. and i.r. spectra;  $^{13}\text{C}$ -n.m.r. data are shown in Figure 1 and i.r. data are shown in Figure 2.

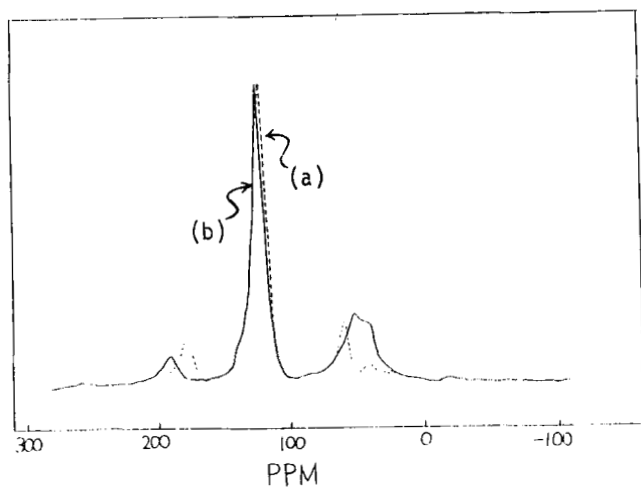


FIGURE 1  $^{13}\text{C}$  n.m.r. spectrum: (a) cis-1,2-dichloroethylene based PVS; (b) trans-1,2-dichloroethylene based PVS.

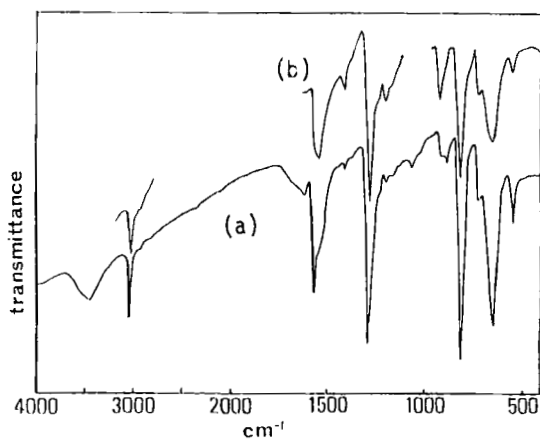


FIGURE 2 i.r. spectrum: (a) cis-1,2-dichloroethylene based PVS; (b) trans-1,2-dichloroethylene based PVS.

The sharp signal at ca. 121.9 ppm for cis-1,2-dichloroethylene based PVS and 122.2 ppm for trans-1,2-dichloroethylene based PVS in  $^{13}\text{C}$ -n.m.r. spectrum indicates the presence of the alkenic carbon atom. The i.r. spectrum exhibits absorption bands at 3024, 1540, 910, 800, 660  $\text{cm}^{-1}$ . The band at 3024  $\text{cm}^{-1}$  is due to the olefinic C-H stretching. A band centered at 1540  $\text{cm}^{-1}$  is characteristic of the C=C double bond positioned between two sulphur atoms, such as  $\text{S}-\text{CH}=\text{CH}-\text{S}^2$ . A sharp band at 800  $\text{cm}^{-1}$  is also characteristic of  $\text{S}-\text{CH}=\text{CH}$  structure. The two bands at 910 and 660  $\text{cm}^{-1}$  can be assigned to the olefinic C-H deformation vibration out of plane in trans and cis conformation, respectively.

The elemental analysis is also supporting the structure of PVS. The result is shown in Table I.

TABLE I Elemental analysis of PVS.

cis- based PVS	C <sub>2.0</sub> H <sub>2.00</sub> S <sub>0.97</sub> Cl <sub>0.032</sub>
trans- based PVS	C <sub>2.0</sub> H <sub>2.16</sub> S <sub>1.02</sub> Cl <sub>0.006</sub>

The molecular ends are thought to be chlorine atoms and from these experimental formula molecular weight can be calculated as 2400 ~ 3300 for cis-1,2-dichloroethylene based PVS and 10000~14000 for trans-1,2-dichloroethylene based PVS respectively.

#### ACKNOWLEDGMENT

This work was performed under the management of the Research Association for Basic Polymer Technology for synthetic metals as a part of a project on Basic Technology for Future Industries sponsored by Agency of Industrial Science and Technology, Ministry of International Trade and Industry.

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