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## Data Article

# $^1\text{H}$ NMR and FT-IR dataset based structural investigation of poly(amic acid)s and polyimides from 4,4'-diaminostilbene



Amit Kumar<sup>a,c,1</sup>, Seiji Tateyama<sup>a,d,1</sup>, Katsuaki Yasaki<sup>a</sup>,  
 Mohammad Asif Ali<sup>a</sup>, Naoki Takaya<sup>b,d</sup>, Rajeev Singh<sup>c</sup>,  
 Tatsuo Kaneko<sup>a,d,\*</sup>

<sup>a</sup> School of Materials Science, Japan Advanced Institute of Science and Technology (JAIST), 1-1 Asahidai, Nomi-shi, Ishikawa 923-1292, Japan

<sup>b</sup> Faculty of Life and Environmental Sciences, University of Tsukuba, Ibaraki 305-8572, Japan

<sup>c</sup> Material/Organometallics Laboratory, Department of Chemistry, ARSD College, University of Delhi, Delhi 110021, India

<sup>d</sup> JST, ALCA, Japan

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## ABSTRACT

Structural investigation of polymers by various available analytical methods is important in order to correlate the structure with polymer properties for which understanding of polymer structure is very important factor. The data presented here in this article shows the  $^1\text{H}$  NMR spectra used for the characterization of prepared poly(amic acid)s (PAAs). It is often difficult to assign the peak in NMR of polymers due to its complexity. Data presented here helps in assigning the proton peak in complex NMR of PAAs prepared from aromatic diamines. Further functionality in polymer chains can be confirmed by FT-IR spectra. Change in functionality during some reaction or process can be monitored by disappearance or appearance of peaks in FT-IR. The complete imidization of PAAs to Polyimides (PIs) is difficult to analyze because of the chemical stability i.e. insolubility of PIs in most of the solvent

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\* Corresponding author at: School of Materials Science, Japan Advanced Institute of Science and Technology (JAIST), 1-1 Asahidai, Nomi-shi, Ishikawa 923-1292, Japan.

E-mail address: [kaneko@jaist.ac.jp](mailto:kaneko@jaist.ac.jp) (T. Kaneko).

<sup>1</sup> Equal contribution to this paper.

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therefore the completion of imidization process was confirmed using FTIR.

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## 1. Specifications Table

Subject area	Chemistry
More specific subject area	Polymer chemistry
Type of data	NMR and FTIR spectra
How data was acquired	NMR and FT-IR. $^1\text{H}$ NMR were performed on a Bruker Biospin AG 400 MHz spectrometer. FT-IR spectra were recorded with a Perkin-Elmer Spectrum One spectrometer between 4000 and $400\text{ cm}^{-1}$ using a diamond-attenuated total reflection (ATR).
Data format	Analyzed
Experimental factors	$^1\text{H}$ nuclear magnetic resonance ( $^1\text{H}$ NMR) was performed using DMSO- $d_6$ as a solvent at 23.1 °C. FT-IR of PAAs and PIs were recorded using thin films.
Experimental features	Poly(amic acid)s (PAAs) are synthesized by a reaction of diamines 4-4' diaminostilbene (DAS) with equimolar amounts of dianhydrides by polycondensation reaction. Polyimide (PI) films were obtained by stepwise thermal imidization of the PAAs films in an oven.
Data source location	School of Materials Science, Japan Advanced Institute of Science and Technology (JAIST), 1-1 Asahidai, Nomi-shi, Ishikawa, 923-1292, Japan 36°26'40.3"N, 136°35'33.5"E 36.444528, 136.592639
Data accessibility	Data is with this article.

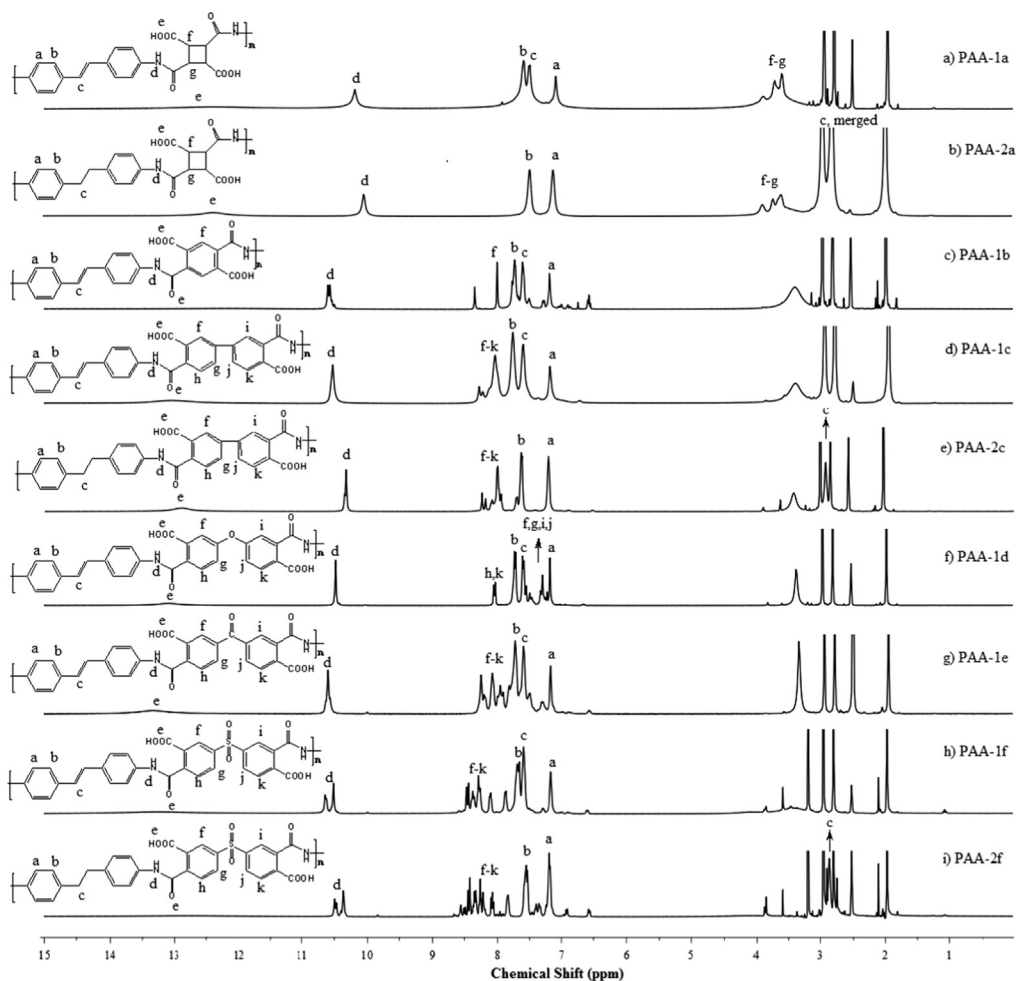
## 2. Value of the data

- This following data helps in assigning the proton peak which is otherwise a complex task in case of polymers.
- The comparison with various other polymers can help in understanding the significant role of aromatic diamines and dianhydride structure in structural-property of polymers.
- The completion of imidization can be easily confirmed by FT-IR by comparing the spectra before and after imidization process.

## 3. Data

In  $^1\text{H}$  NMR spectra (Fig. 1) of PAAs derived with 4-4'diaminostilbene (DAS), the main chain proton signals for amides, aromatics proton of dianhydride, aromatic proton of diamine DAS and  $-\text{CH}=\text{CH}-$  appeared around 10.7–10.1, 8.6–7.3, 7.6–7.1 and 7.6–7.4 ppm respectively. In case of 4,4'-(ethane-1,2-diy)dianiline (EDDA) derived PAAs,  $-\text{CH}=\text{CH}-$  protons peaks was replaced by proton peak of  $-\text{CH}_2-\text{CH}_2-$  at 2.8–2.7 ppm however other peaks remained unaltered.

Figs. 2 and 3 show the FT-IR spectra of the PAAs and PIs respectively. In all the samples the following peaks were observed: broad band in the range  $2500\text{--}3500\text{ cm}^{-1}$  (O–H stretching of carboxylic acid group's hydroxyls), two different carbonyl peaks at  $1720\text{ cm}^{-1}$  (C=O stretching, carboxylic) and

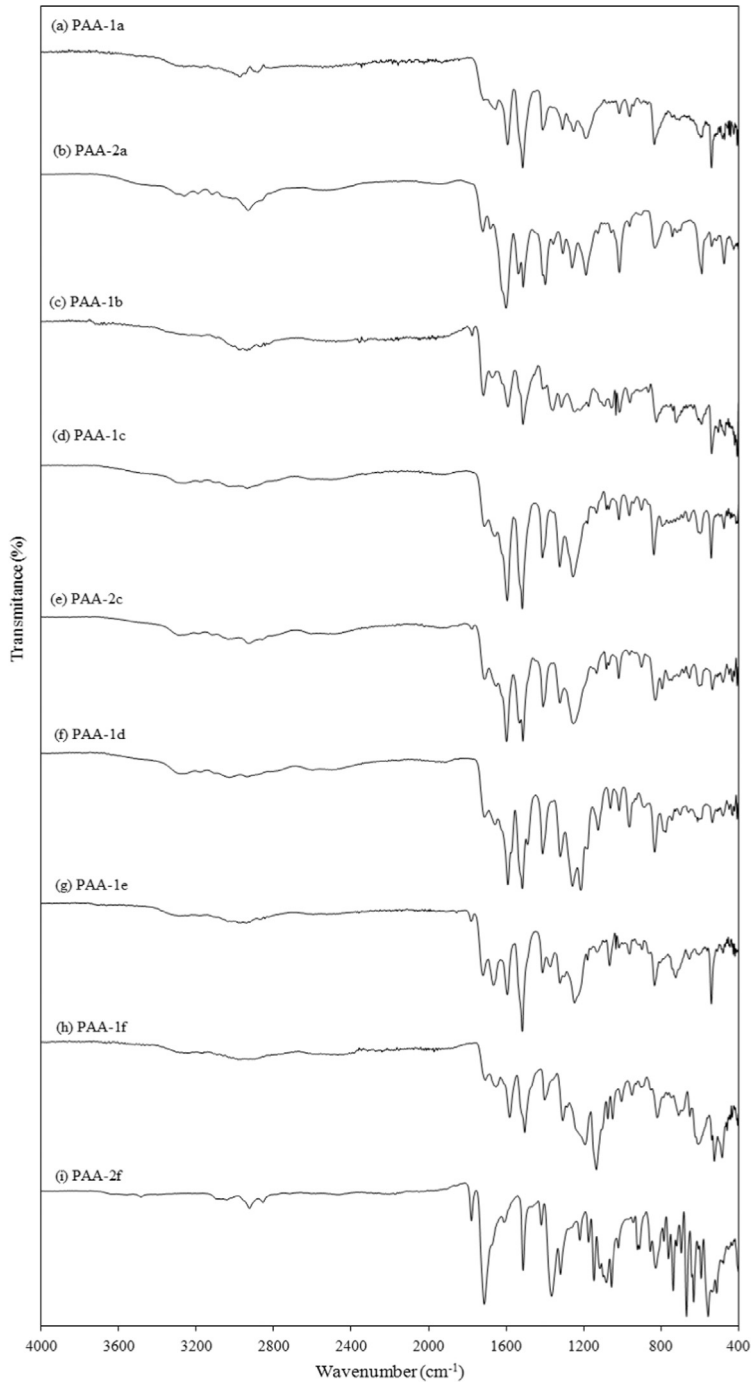


**Fig. 1.**  $^1\text{H}$  NMR spectra of (a) PAA-1a, (b) PAA-2a, (c) PAA-1b, (d) PAA-1c, (e) PAA-2c, (f) PAA-1d, (g) PAA-1e, (h) PAA-1f and (i) PAA-2f.

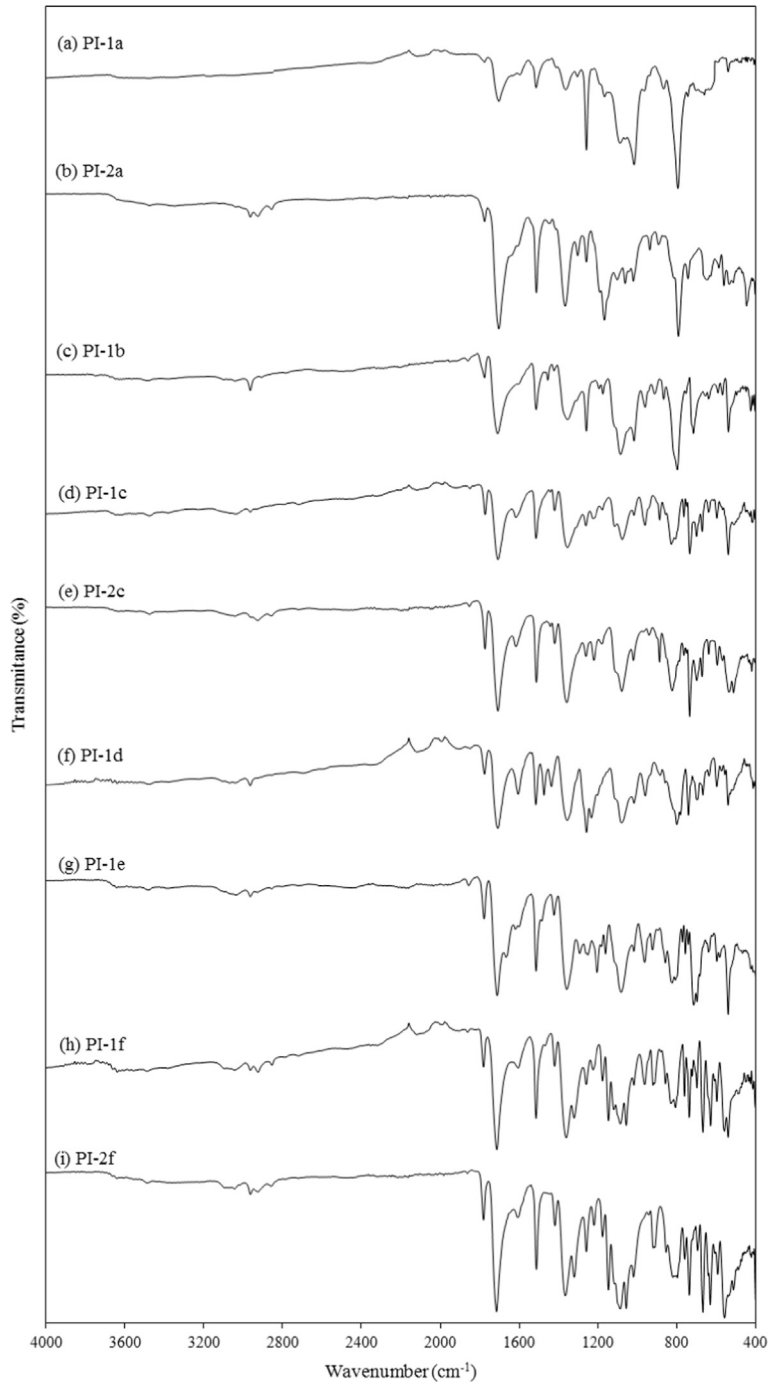
$1670\text{ cm}^{-1}$  ( $\text{C}=\text{O}$  stretching, amide), and  $1520$  and  $1430\text{ cm}^{-1}$  (aromatic  $\text{C}-\text{H}$  overtone aromatic). After imidization  $1720\text{ cm}^{-1}$  ( $\text{C}=\text{O}$  stretching, carboxylic) and  $1670\text{ cm}^{-1}$  ( $\text{C}=\text{O}$  stretching, amide) were replaced by a small peak at  $1780\text{ cm}^{-1}$  ( $\text{C}=\text{O}$  asymmetric stretching) and a big peak at  $1712\text{ cm}^{-1}$  ( $\text{C}=\text{O}$  symmetric stretching) which were characteristic to PI structures. Furthermore, other peaks at  $1518\text{ cm}^{-1}$  ( $\text{C}-\text{C}$  stretching of aromatic),  $1441\text{ cm}^{-1}$  ( $\text{C}=\text{C}$  stretching of p-substituted benzene)  $1376\text{ cm}^{-1}$ , ( $\text{C}-\text{N}$  stretching of imide), and  $1175\text{ cm}^{-1}$  (imide ring deformation) appeared, which indicated a complete imidization [1]. OPDA derived PI-1d showed IR peak at  $1235\text{ cm}^{-1}$  corresponding to  $\text{C}-\text{O}$  stretching of ether group and DSPDA derived PI-1f and PI-2f showed asymmetric and symmetric  $\text{S}=\text{O}$  stretching at  $1323$  and  $1148\text{ cm}^{-1}$  respectively [2].

#### 4. Experimental design, materials and methods

For NMR  $\text{DMSO}-d_6$  was used as a solvent at  $23.1\text{ }^\circ\text{C}$  with 16 accumulation scans, using the proton resonance of residual non-deuterated DMSO as an internal standard (2.55 ppm). PAAs and PIs film of



**Fig. 2.** FT-IR spectra of (a) PAA-1a, (b) PAA-2a, (c) PAA-1b, (d) PAA-1c, (e) PAA-2c, (f) PAA-1d, (g) PAA-1e, (h) PAA-1f and (i) PAA-2f.



**Fig. 3.** FT-IR spectra of (a) PI-1a, (b) PI-2a, (c) PI-1b, (d) PI-1c, (e) PI-2c, (f) PI-1d, (g) PI-1e, (h) PI-1f and (i) PI-2f.

thickness 30–45  $\mu\text{m}$  was used for FT-IR analysis. PAA film of thickness 30–45  $\mu\text{m}$  was obtained by dissolving polymer fibrils in DMAc/DMF forming a yellow solution, poured on a silicon wafer/aluminum plate by spin coating and dried by heating over hot plate. Polyimide (PI) films were obtained by stepwise thermal imidization of the PAAs film in an oven for 1 h each at 100, 150, 200 and 250  $^{\circ}\text{C}$ , respectively, under vacuum [3].

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## Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at <http://dx.doi.org/10.1016/j.dib.2016.02.006>.

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