Arabian Journal of Chemistry (2016) 9, S1265–S1271

King Saud University

Arabian Journal of Chemistry

www.ksu.edu.sa
www.sciencedirect.com

REVIEW

Synthesis and thermal study of phosphorus containing dendron using 2-butyne-1,4-diol at the core

E. Dadapeer, K. Reddi Mohan Naidu, C. Naga Raju *

Department of Chemistry, Sri Venkateswara University, Tirupati 517 502, India

Received 10 September 2011; accepted 7 February 2012
Available online 15 February 2012

KEYWORDS

Phosphorus dendrimer;
Divergent build-up;
Condensation reaction;
2-Butyne-1,4-diol;
Schiff's base;
SEM;
TGA/DTA analysis;
2-Mercaptoethanol

Abstract Synthesis of new phosphorus dendron containing 2-butyne-1,4-diol at the core is described. The divergent build-up of the dendron is based on simple condensation reactions performed by using $P(O)Cl_3$, 3-hydroxybenzaldehyde, hydrazine hydrate, 4-hydroxybenzaldehyde and 2-mercaptoethanol. The final dendron is a Schiff's base macromolecule possessing 8 imine bonds and 8 OH groups at the periphery. The intermediate dendrons were confirmed by IR, NMR (^1H, ^{13}C and ^{31}P), LC–Mass and C, H, N analyzes. The structure of the final dendron **6** was confirmed by IR, NMR (^1H, ^{13}C and ^{31}P), MALDI-TOF-MS, and C, H, N analysis. The surface morphology of the dendron **6** was understood by Scanning Electronic Microscopic study (SEM). To determine the changes in weight in relation to change in temperature of the final dendron **6** was studied by TGA/DTA analysis.

Contents

---

* Corresponding author. Tel.: +91 9703193375.
E-mail address: rajuchamarthi10@gmail.com (C. Naga Raju).

Peer review under responsibility of King Saud University.

Production and hosting by Elsevier

http://dx.doi.org/10.1016/j.arabjc.2012.02.001

1. Introduction

The field of dendrimer chemistry (Zimmerman, 1999) has undergone a rapid development in establishing the synthetic approaches, characterization of dendrimer properties and design of functional dendrimers (Archut and Vögtle, 1998). The hyperbranched and monodispersed macromolecules which are commonly referred to as dendrimers are becoming important in efficient nanomaterials for a variety of applications in medicine, catalysis and electronics (Antoni et al., 2007). Nowadays development of chemistry of dendrimers is becoming an attractive field of research in nanosciences (Newkome and Shreiner, 2008). In general, they consist of core, branching structure and periphery. So, the physical and chemical properties and hence the applications of the dendrimers depend on the nature of these basic constituents. The dendrimers containing heteroatom occupy a relatively marginal place when compared to organic dendrimers, but a rich diversity of structures is available, particularly for phosphorus dendrimers and they possess interesting properties (Majoral and Caminade, 1999). The presence of phosphorus on the surface or within the cascade structure of dendrimer or at the focal point confers to phosphorus dendrimers fascinating properties. The very first example of phosphorus-containing dendrimer was polyphosphonium dendrimer (Rengan and Engel, 1990) and the first neutral phosphorus dendrimer was described by Launay et al. (1994). The phosphorus dendrimers possess interesting and unprecedented properties, which led to some potential applications from biology to material science (Majoral and Caminade, 2003). A few properties of P-dendrimers were highlighted by Caminade et al.(2010). A significant effort in the design and synthesis of these dendritic architectures has led to inside-out (divergent) or outside-in (convergent) methodologies (Washio et al., 2005). The synthetic elaboration and study of the properties of dumbbell-shaped dendrimers are much less explored when compared with globular dendrimers (Tatiana and Ashok, 2008). We have reported (Dadapeer et al., 2010) a novel linear phosphorus containing dendron using diphenylsilanediol at the core. In this paper we intrigued by the possibility of developing a dendron using 2-butyne-1,4-diol at the core giving rise to linear dumbbell-shape by using divergent method. We focus on scanning electron microscopic study to know in particular how the nanometric size property to be considered and thermogravimetric analysis to show the thermal stability and change in weight in relation to change in temperature for the final dendron **6**.

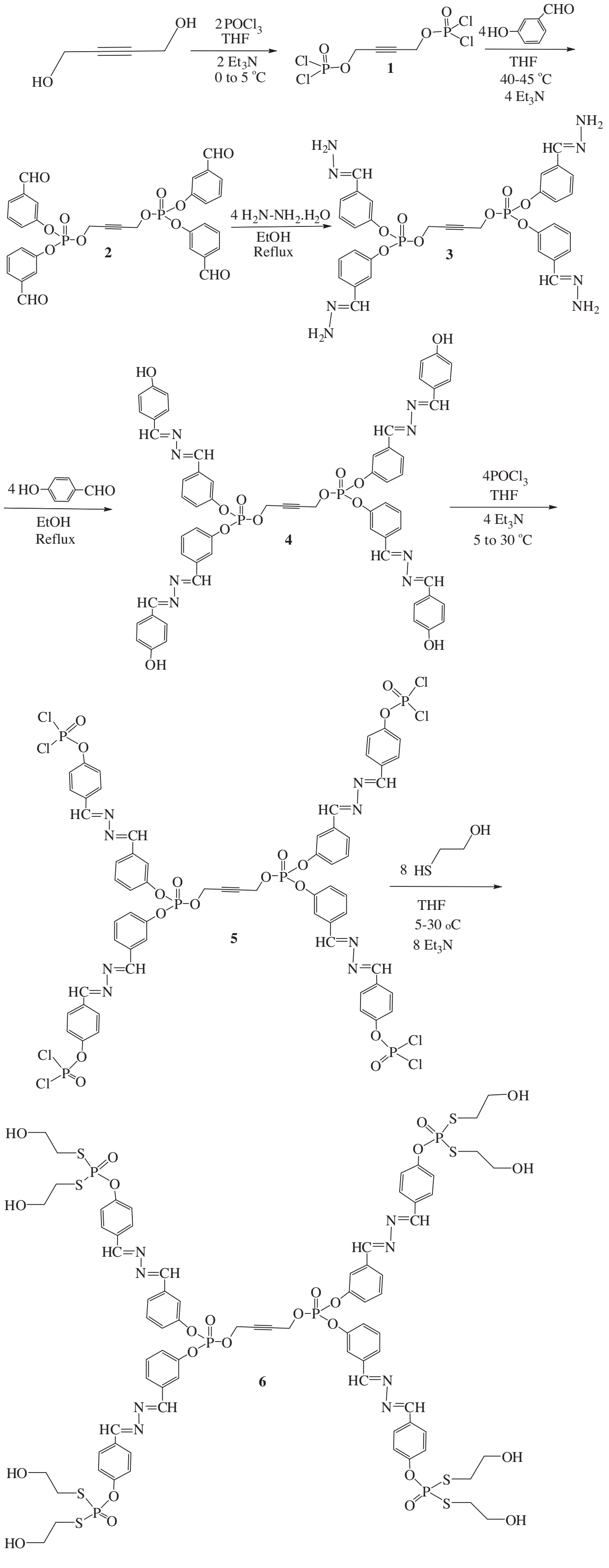
2. Experimental section

2.1. Materials and reagents

All the reagents used in this study were purchased from Sigma–Aldrich chemical company and used without further purification. THF and EtOH were dried by standard method. TLC was performed on precoated plates with silica gel $60F_{254}$ (Merk). Column chromatography was performed on silica gel (0.040–0.063 mm, Macherey, Nagel).

2.2. Instruments

IR Spectra were recorded on JASCO (Japan) FT-IR-5300 spectrometer at the University of Hyderabad using KBr (disk). ^{1}H and ^{13}C NMR spectra of the final dendron were recorded on a Bruker A VIII 500 MHz NMR spectrometer at IIT, Chennai, operating at 500.13 MHz for ^{1}H, 125.75 MHz for ^{13}C NMR, data were recorded in DMSO-d_6 and chemical shifts were referenced to TMS. ^{1}H and ^{13}C NMR spectra of the intermediate dendrons were recorded on a Bruker A VIII 400 MHz NMR spectrometer at Laila Impex, Vizayawada, India, operating at 400.13 MHz for ^{1}H, 100.61 MHz for ^{13}C NMR, data were recorded in DMSO-d_6 and chemical shifts were referenced to TMS. ^{31}P NMR was recorded on Bruker ACF Supercon 200 spectrometer operating at 81 MHz at the University of Hyderabad, Hyderabad. ^{31}P NMR data were recorded in $CDCl_3$ or DMSO-d_6 and chemical shifts were referenced to 85% H_3PO_4. EI Mass spectra of intermediate compounds were recorded on JEOL GC mate at IIT, Chennai. MALDI mass spectrum of the final dendron **6** was recorded using Applied Biosystems MALDI-TOF Voyager depro spectrometer. The sample was run using Sinapic acid as the matrix with DMSO as the solvent in dried-droplet preparation method, performed at IIT-Madras, Chennai. TGA–DTA measurement was taken using TA instrument, Waterloo, USA, performed at Sri Krishnadevaraya University, Ananthapur, India. Simultaneous TGA–DTA measures both heat flow



Scheme 1 The schematization of the synthesis of the esthetic structure of dumbbell-shaped dendron emanating radially from a central core.

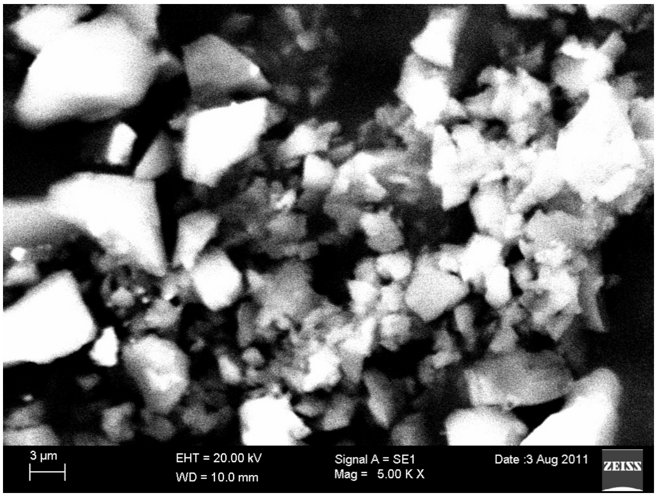


Figure 1 SEM photograph of the dendron **6**.

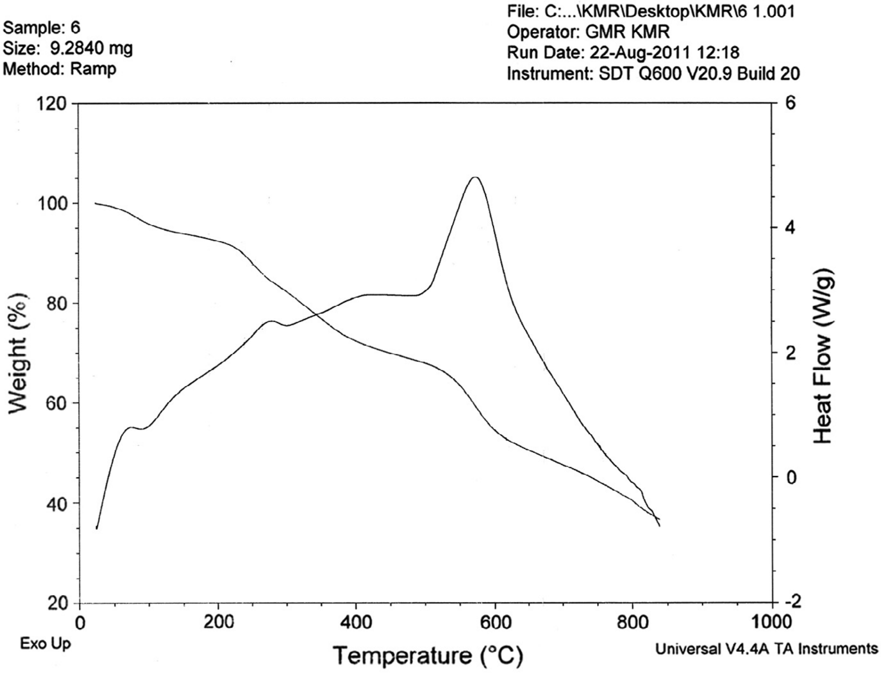


Figure 2 TGA–DTA of dendron **6**.

and weight changes (TGA) in a material as a function of temperature in a controlled air atmosphere were recorded. SEM operated at 20 kV, performed at the Department of Physics, S.V. University, Tirupati, India. Elemental analyzes were performed using EA 1112 Thermo Finnigan instrument, France, at the University of Hyderabad, Hyderabad, India.

2.3. General procedure for synthesis of dendron ***6***

2.3.1. Preparation of ***1***

A solution of $POCl_3$ (0.01 mol, 0.93 mL) in 20 mL of dry THF was added dropwise over a period of 20 min to a mixture of stirred solution of 2-butyne-1,4-diol (0.005 mol, 0.43 g) in 25 mL of dry THF and triethylamine (0.01 mol, 1.4 mL) at −5 to 0 °C. After stirring for 3 h at 35–40 °C, formation of **1** was ascertained by TLC analysis run in a 3:7 mixture of ethyl acetate and hexane. Triethylamine hydrochloride was removed by filtration. The filtrate was used for the next reaction step without further purification. The compound thus obtained was characterized by IR, NMR (^{1}H, ^{13}C and ^{31}P), LC–Mass and C, H, N analyzes and used for the next synthetic step. The average R_f value observed was 0.70. Yield: 80% (solid); IR (KBr, cm^{-1}): 1232 (P=O str), 1072 (P–O–C_{aliph} str); ^{1}H NMR (500 MHz, DMSO-d_6, δ/ppm): 4.42–4.56 (4H, m, –CH_2–O–); ^{13}C NMR (100 MHz, DMSO-d_6, δ/ppm): 86.3, 53.5; ^{31}P NMR (81 MHz, $CDCl_3$, δ /ppm): −1.72; EI Mass:

m/*z* 319.58 $[M]^+$; Anal. Calcd for $C_4H_4Cl_4O_4P_2$: C, 15.02; H, 1.26; Found: C, 15.05; H, 1.29.

2.3.2. Preparation of **2**

To a mixture of stirred solution of **1** and triethylamine (0.02 mol, 2.8 mL), a solution of 3-hydroxybenzaldehyde (0.02 mol, 2.44 g) in 25 mL of dry THF was added dropwise over a period of 20 min at 5–15 °C. After stirring for 4 h at 40–45 °C, formation of **2** was ascertained by TLC analysis run in a 4:6 mixture of ethyl acetate and hexane. Triethylamine hydrochloride was removed by filtration. The solvent was evaporated under reduced pressure to give crude product. It was purified by column chromatography on silica gel (60–120 mesh, ethyl acetate:hexane, 2:8) to afford the pure compound. The compound thus obtained was characterized by IR, NMR (^{1}H, ^{13}C and ^{31}P), LC–Mass and C, H, N analyzes and used for the next synthetic step. The average R_f value observed was 0.60. Yield: 70% (solid); IR (KBr, cm^{-1}): 1693 (CHO stretching), 1236 (P=O str), 931, 1167 (P–O–C_{arom}), 1076 (P–O–C_{aliph}); ^{1}H NMR (500 MHz, DMSO-d_6, δ/ppm): 9.90 (4H, s, CHO), 7.16–7.37 (16H, m, Ar-H), 3.92–3.97 (4H, m, –CH_2–O–); ^{13}C NMR (125 MHz, DMSO-d_6, δ/ppm): 192.9, 158.4, 137.6, 129.9, 121.8, 120.5, 115.0, 86.0, 52.5; ^{31}P NMR (81 MHz, 85% H_3PO_4, δ/ppm): −13.93; EI Mass: *m*/*z* 662.20 $[M]^+$; Anal. Calcd for $C_{32}H_{24}O_{12}P_2$: C, 58.02; H, 3.65; Found: C, 58.04; H, 3.68.

2.3.3. Preparation of **3**

To a stirred solution of **2** in 25 mL of dry EtOH, a solution of hydrazine hydrate (0.02 mol, 1 mL) in 25 mL of dry EtOH, was added at room temperature. After stirring for 5 h at reflux temperature, formation of **3** was ascertained by TLC analysis run in a 1:1 mixture of ethyl acetate and hexane. The solvent was evaporated under reduced pressure to give crude product. It was purified by washing with ethyl acetate and hexane. The imine thus obtained was characterized by IR, NMR (^{1}H, ^{13}C and ^{31}P), LC–Mass and C, H, N analyzes and used for the next synthetic step. The average R_f value observed was 0.65. Yield: 65% (solid); IR (KBr, cm^{-1}): 3192 (str), 1452 (bending) (–NH_2), 1583 (CH=N), 985, 1155 (P–O–C_{arom}), 1232 (P=O), 1056 (P–O–C_{aliph}); ^{1}H NMR (500 MHz, DMSO-d_6, δ/ppm): 10.41 (4H, s, –CH=N), 6.88–7.78 (16H, m, Ar-H), 3.78–3.89 (4H, m, –CH_2–O–), 2.9 (8H, s, –NH_2); ^{13}C NMR (125 MHz, DMSO-d_6, δ/ppm): 160.9, 159.7, 136.5, 130.1, 124.7, 120.2, 115.8, 79.2, 57.0; ^{31}P NMR (81 MHz, 85% H_3PO_4, δ /ppm): −1.50; EI Mass: *m*/*z* 719.61 $[M+1]^+$; Anal. Calcd for $C_{32}H_{32}N_8O_8P_2$: C, 53.49; H, 4.49; N, 15.59; Found: C, 53.48; H, 4.47; N, 15.61.

2.3.4. Preparation of **4**

To a stirred solution of **3** in 30 mL of dry EtOH, a solution of 4-hydroxybenzaldehyde (0.02 mol, 2.44 g) in 25 mL of dry EtOH, was added at room temperature. After stirring for 5 h at reflux temperature, formation of **4** was ascertained by TLC analysis run in a 4:6 mixture of ethyl acetate and hexane. The solvent was evaporated under reduced pressure to get crude product. It was purified by washing with ethyl acetate and hexane. The imine thus obtained was characterized by IR, ^{1}H, ^{13}C, ^{31}P NMR, MALDI-TOF-MS and C, H, N analyzes. The average R_f value observed was 0.65. Yield: 60% (solid); IR (KBr, cm^{-1}): 3349 (Ar-OH), 1590 (CH=N), 963, 1165 (P–O–C_{arom}), 1228 (P=O), 1053 (P–O–C_{aliph}); ^{1}H NMR (500 MHz, DMSO-d_6, δ/ppm): 9.78, 9.91 (8H, s, –CH=N), 8.58 (4H, br, Ar-OH), 6.85–7.76 (32H, m, Ar-H), 4.00–4.05 (4H, m, –CH_2–O–); ^{13}C NMR (125 MHz, DMSO-d_6, δ/ppm): 161.9, 160.8, 158.1, 147.4, 135.5, 130.5, 130.4, 125.5, 122.0, 120.4, 116.3, 114.5, 84.0, 59.0. ^{31}P NMR (81 MHz, 85% H_3PO_4, δ /ppm): −7.45; MALDI mass: *m*/*z* 1135.5 $[M^+]$; Anal. Calcd for $C_{60}H_{48}N_8O_{12}P_2$: C, 63.49; H, 4.26; N, 9.87; Found: C, 63.52; H, 4.29; N, 9.89.

2.3.5. Preparation of **5**

To a mixture of stirred solution of the filtrate **4** and triethylamine (0.02 mol, 2.8 mL), a solution of $POCl_3$ (0.02 mol, 1.66 mL) in 20 mL of dry THF was added dropwise over a period of 20 min at 25 °C. After stirring for 5 h at 40–45 °C, formation of **5** was ascertained by TLC analysis run in a 2:4 mixture of ethyl acetate and hexane. Triethylamine hydrochloride was removed by filtration. It was purified by column chromatography on silica gel (60–120 mesh, ethyl acetate:hexane, 2:8) to afford the pure compound. The compound thus obtained was characterized by IR, NMR (^{1}H, ^{13}C and ^{31}P), LC–Mass and C, H, N analyzes and used for the next synthetic step. The average R_f value observed was 0.57. Yield: 57% (solid); IR (KBr, cm^{-1}): 1612 (CH=N), 954, 1187 (P–O–C_{arom}), 1245 (P=O), 1061 (P–O–C_{aliph}); ^{1}H NMR (500 MHz, DMSO-d_6, δ/ppm): 9.78, 9.91 (8H, s, –CH=N), 6.25–7.68 (32 H, m, Ar-H), 4.15–4.20 (4H, m, –CH_2–O–); ^{13}C NMR (125 MHz, DMSO-d_6, δ/ppm): 60.1, 85.5, 159.2, 115.0, 135.1, 121.0, 129.3, 121.8, 148.0, 162.2, 126.1, 129.5, 117.2, 161.1; ^{31}P NMR (81 MHz, 85% H_3PO_4, δ /ppm): −10.50, −8.44; LC–Mass: *m*/*z* 1600 $[M-1]^+$; Anal. Calcd for $C_{60}H_{44}Cl_8N_8O_{16}P_6$: C, 44.53; H, 2.77; N, 6.99; Found: C, 44.51; H, 2.79; N, 7.01.

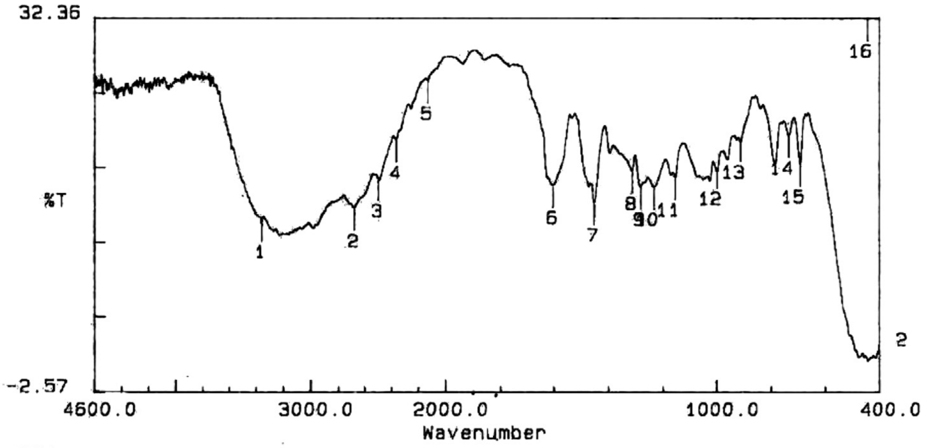
2.3.6. Preparation of **6**

To the mixture of stirred solution of **5** and triethylamine (0.04 mol, 5.6 mL), a solution of 2-mercaptoethanol (0.04 mol, 2.81 mL) in 20 mL of dry THF was added dropwise at 10 °C. After stirring for 5 h at 30 °C, formation of **6** was ascertained by TLC analysis run in a 1:1 mixture of ethyl acetate and hexane. Triethylamine hydrochloride was removed by filtration. The product was purified by column chromatography on silica gel (60–120 mesh, ethyl acetate:hexane, 7:3) to afford the pure compound. The compound thus obtained was characterized by IR, NMR (^{1}H, ^{13}C and ^{31}P), MALDI-TOF-MS and C, H, N analyzes. The average R_f value observed was 0.50. Yield: 50% (solid); IR (KBr, cm^{-1}): 3280 (–OH), 1622 (CH=N), 951, 1176 (P–O–C_{arom}), 1240 (P=O), 1055 (P–O–C_{aliph}); ^{1}H NMR (500 MHz, DMSO-d_6, δ/ppm): 9.81, 9.95 (8H, s, –CH=N), 6.65–7.98 (32 H, m, Ar-H), 4.55–4.85 (4H, m, –CH_2–O–), 3.58 (8H, s, –OH), 3.55 (16H, t, J = 8 Hz, S–CH_2–CH_2–O), 2.56 (16H, t, J = 10 Hz, S–CH_2–CH_2–O); ^{13}C NMR (125 MHz, DMSO-d_6, δ/ppm): 162.1, 161.1159.7, 147.7, 131.1, 136.2, 129.9, 126.1, 121.9, 121.6, 115.8, 115.5, 85.2, 58.8; ^{31}P NMR (81 MHz, 85% H_3PO_4, δ/ppm): −7.12, −1.88; MALDI mass: *m*/*z* 1934.95 $[M^+]$; Anal. Calcd for $C_{76}H_{84}N_8O_{24}P_6S_8$: C, 47.15; H, 4.37; N, 5.79; Found: C, 47.18; H, 4.39; N, 5.80.

3. Results and discussion

3.1. Chemistry

The synthetic strategy of the esthetic structure of dumbbell-shaped novel phosphorus containing dendron **6** emanating radially from 2-butyne-1,4-diol central core is shown in Scheme 1. The 2-butyne-1,4-diol was used as linear core and dendrons are built around it. For the synthesis of dendron **1**, a condensation reaction of 1molar equivalent of 2-butyne-1,4-diol with 2 equivalents of $POCl_3$ in the presence of triethylamine at −5 to 40 °C in dry THF with stirring for 2–3 h afforded compound **1**. After filtration, **1** was then treated with 4 equivalents of 3-hydroxybenzaldehyde in dry THF in the presence of triethylamine at 40–45 °C with stirring for 5 h to form **2**. After filtration, compound **2** was reacted with 4 equivalents of hydrazine hydrate with stirring for 5 h under reflux in dry EtOH to afford **3**. The compound **3** was treated with 4-hydroxybenzaldehyde in refluxing dry EtOH with stirring for 6 h to afford **4**. Then compound **4** was treated with 4 equivalents of $POCl_3$ in the presence of triethylamine at 5–30 °C in dry THF with stirring for 5 h to yield **5**. Finally, compound **5** was treated with 8 equivalents of 2-mercaptoethanol in the presence of triethylamine at −15 to 30 °C in dry THF with stirring for 6 h to obtain **6**. As the size of the dendron increases, the reaction is sluggish and the yield is low. To improve the yield of **6**, solvent is taken in excess and appropriate temperature is maintained and stirring was continued for long periods. Synthesis of the dendron **6** was accomplished by adopting divergent approach as shown in Scheme 1. The overall yield of the final dendron **6** was 50%. The formation of all the intermediate compounds **1**, **2**, **3**, **4** and **5** was characterized by IR, NMR (^1H, ^{13}C and ^{31}P), LC–Mass, MALDI-TOF-MS and C, H, N analyzes. The synthetic and analytical data of the dendron **6** and the intermediate compounds **1**, **2**, **3**, **4** and **5** are given in the experimental part. The dendron **6** exhibited absorption bands for –OH, P=O, in the regions 3280, 1240 cm^{-1}, respectively. $P–O–C_{arom}$ gave two absorption bands in the regions 951 and 1176 cm^{-1} (Thomas, 1974). $P–O–C_{aliph}$ gave absorption in the region 1015 cm^{-1}(Thomas, 1974). Absorption band for CH=N was observed at 1622 cm^{-1}. In the ^1H NMR spectra (500 MHz) of **6**, the aromatic protons gave multiplet in the region δ 6.65–7.98. The –OH protons resonated at δ 3.58 as singlet. The CH=N protons gave chemical shifts in the region δ 9.81 and 9.95 in two environments. The $–\underline{CH_2}–O–$ protons resonated as singlet at δ 4.55–4.85. ^{13}C NMR spectrum was recorded for **6** and the data are given in the experimental part. The aromatic carbons resonated in the region 115.5–159.7 ppm. Carbon signal for $–\underline{CH}=N$ was observed at δ 162.1. The **6** gave two ^{31}P NMR signals at δ −7.12, −1.88. The intermediate compounds **1**, **2**, **3**, **4** and **5** showed phosphorus-31 resonance signals in the expected regions and their data are given in the experimental section. The molecular weight of the final dendron **6** was determined by Matrix Assisted Laser Desorption/Ionization (MALDI-TOF) mass spectrometry. The mass obtained from the MALDI measurements corresponds closely to the calculated value. The surface topography of dendron **6** was studied by Scanning Electron Microscopy (SEM). The molecular decomposition of the dendron **6** was investigated by Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA).



```
Condition
   upper  32.35     lower -2.57     depth    1.00
Peak table
 1:   3280.48 ( 12.1)     2:   2679.37 ( 14.6)     3:   2499.97 ( 17.4)     4:   2364.94 ( 21.0)
 5:   2129.60 ( 26.5)     6:   1622.30 ( 15.2)     7:   1452.53 ( 15.2)     8:   1313.64 ( 18.1)
 9:   1282.78 ( 16.6)    10:   1240.62 ( 16.6)    11:   1176.46 ( 17.4)    12:   1015.22 ( 17.5)
13:    951.22 ( 19.4)    14:    734.95 ( 21.4)    15:    692.51 ( 18.7)    16:    443.67 (  0.3)
```

IR spectrum of compound 6

3.2. Scanning electronic microscopic study

Deeper insight surface structural characteristics of the dendron **6** were studied by Scanning Electron Microscopy (SEM). With increasing the size it was clear that the overall surface morphology of the material tended to assume a gravel shape at a line width of 3 µm. Fig. 1 depicts the surface structure of the dendrimer **6** having 8 OH groups on the periphery.

3.3. TGA–DTA analysis

An overlay of TGA and DTA plots for the dendron **6** up to 850 °C in air atmosphere is shown in Fig. 2. The decomposition of the final dendron **6** was followed by TGA. An initial decomposition starts in between the range of 80–90 °C with a corresponding weight loss of approximately 2%. This is attributed to the loss of water molecules. Then immediately decomposition starts in between the range of 100–230 °C with a corresponding weight loss of approximately 8–10%. Then dendron **6** is again decomposed in between the temperature range of 250–525 °C with a corresponding weight loss of approximately 23–25%. Then immediately starts the decomposition in between the range of 530–600 °C with a corresponding weight loss of approximately 10–12%. The total account for the loss of dendron **6** is around 50% of the total mass of the molecule. Again the dendron **6** is slowly decomposed and continued up to 850 °C. Similarly, Differential Thermal Analysis (DTA) is also tested on dendrimer **6** to detect the changes in the sample, either exothermic or endothermic. The DTA profile (Fig. 2) shows the endotherm peak at 85°C corresponds to the loss of water and an exotherm peak at 375°C, corresponds to the approximate 35% loss. Remaining exothermic peaks around at 410 and 590 °C are connected with the loss of remaining percent of dendritic wedges of **6**.

4. Conclusion

Synthesis of a novel phosphorus dendron **6** was accomplished by divergent approach. The condensation reactions were performed in dry THF in the presence of triethylamine and the Schiff's base preparations were performed in dry ethanol. The surface topography of dendron **6** was observed by SEM image. It was tended to assume a nanoscale size gravel shape at a line width of 3 μm. The thermal stability and changes in weight in relation to change in temperature and the heat of the final dendritic molecule **6** were established by TGA–DTA analysis. It reveals that the dendron **6** starts decomposition in the range of 100–230 °C and continues up to 530–600 °C with a corresponding weight loss of approximately 50% of weight loss. About 2% of water loss takes place at around 80–90 °C. The DTA curve reveals that the heat change is endothermic for the loss of water at 85 °C and an exotherm peak at 375 °C, corresponds to the approximate 35% loss. Remaining exothermic peaks around at 410 and 590 °C are connected with the loss of remaining percent of dendritic wedges of **6**.

Acknowledgment

The authors are grateful to HRDG, the CSIR, New Delhi, India, for sanctioning a major research project (01(2309)/09/EMR-II).

References

Antoni, P., Nystrom, D., Hawker, C.J., Hulta, A., Malkoch, M., 2007. Chem. Commun., 2249.
Archut, A., Vögtle, F., 1998. Chem. Soc. Rev. 27, 233.
Caminade, A.M., Laurent, R., Turrin, C.O., Rebout, C., Nicot, B.D., Ouali, O., Majoral, J.P., 2010. C. R. Chim. 13, 1006.
Dadapeer, E., Hari Babu, B., Suresh Reddy, C., Naga Raju, C., 2010. Beilstein J. Org. Chem. 6, 726.
Launay, N., Caminade, A.M., Lahana, R., Majoral, J.P., 1994. Angew. Chem., Int. Ed. Engl. 33, 1589.
Majoral, J.P., Caminade, A.M., 1999. Chem. Rev. 99, 845.
Majoral, J.P., Caminade, A.M., 2003. Top. Curr. Chem. 223, 111.
Newkome, G.R., Shreiner, C.D., 2008. Polymer 49, 1.
Rengan, K., Engel, R., 1990. J. Chem. Soc., Chem. Commun. 16, 1048.
Tatiana, V., Ashok, K., 2008. Can. J. Chem. 86, 540.
Thomas, L.C., 1974. The Interpretation of Infrared Spectra of Organophosphorus Compounds. Heydon, London, New York.
Washio, I., Shibasaki, Y., Mitsuru, U., 2005. Macromolecules 38 (6), 2237.
Zimmerman, S.C., 1999. J. Chem. Educ. 76 (1), 31.