

Bioconjugate Chemistry

Bioconjugate Chem., 1998, 9(6), 645-654, DOI:[10.1021/bc970199d](https://doi.org/10.1021/bc970199d)

Terms & Conditions

Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at <http://pubs.acs.org/page/copyright/permissions.html>



ACS Publications

MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1998 American Chemical Society

Synthesis of Water Soluble, Non-immunogenic Polyamide Cross-linking Agents

Ton That Hai, D. Pereira, and D. Nelson

Hemoglobin Therapeutics Division, Baxter Healthcare Corp., Round Lake, IL 60073 USA

Supporting Information.

Table S-1. NMR Data for Polyamide Precursors

Polyamide	¹ H-NMR Chemical Shifts	¹³ C-NMR Chemical Shifts
1a	(In DMSO-d ₆) 1.40 (br. s, 2H), 2.61 (br. s, 2H), 3.95 (s, 2H), 4.13 (s, 2H), 7.89 (s, 1H), 12.81 (s, 1H) ppm.	(In DMSO-d ₆) 26.7, 38.1, 68.0, 70.8; 168.8, 172.1 ppm.
10a	(In CDCl ₃) 1.52 (s, 2H), 3.25 (m, 2H), 3.70 (s, 3H), 3.99 (s, 2H), 4.08 (s, 2H), 6.98 (s, 1H) ppm.	(In CDCl ₃) 26.6, 38.3, 51.9, 68.4, 71.2; 168.8, 170.2 ppm.
10b	(In DMSO-d ₆) 1.38 (br.s, 2H), 3.10 (br s, 2H), 4.18 (s, 2H), 4.52 (s, 2H), 7.48 (d, 2H, J = 9.0 Hz), 7.85 (t, 1H, J = 5.6 Hz), 8.30 (d, 2H, J = 9.0 Hz) ppm.	(In DMSO-d ₆) 26.6, 37.9, 67.8, 70.1, 123.1, 125.4, 126.2, 145.2, 152.0, 154.9, 168.4 ppm.
10d	(In DMSO-d ₆) 1.39 (br.s, 2H), 3.10 (br s, 2H), 3.94 (s, 2H), 4.09 (s, 2H), 7.21 (m, 3H), 7.48 (m, 1H), 7.85 (t, 1H, J = 5.7 Hz) ppm.	(In DMSO-d ₆): 26.6, 37.9, 67.9, 70.2, 110.1, 123.9, 125.2, 148.4, 168.7, 171.5 ppm.
14b	(In CDCl ₃): 3.07 (t, 2H, J = 6.8 Hz), 3.89 (t, 2H, J = 6.8 Hz), 7.32 (m, 3H), 8.08 (m, 1H) ppm.	(In CDCl ₃): 22.7, 39.9, 109.6, 116.4, 125.5, 126.2, 129.6, 146.4, 172.2, 178.5 ppm.
9	(In CDCl ₃) 3.55 (s, 3H), 6.61 (m, 1H), 7.08 (t, 1H, J = 2.5 Hz), 7.15-7.50 (m, 9H) ppm.	(In CDCl ₃): 34.7, 117.7 (d,), 119.8 (d,), 120.2 (d,), 120.3., 129.5, 149.2 (d,), 166.4 (d,) ppm. ³¹ P-NMR (CDCl ₃): -15.2 ppm.
14c	(In CDCl ₃): 3.00 (t, 2H, J = 6.9 Hz), 3.57 (s, 3H), 3.92 (t, 2H, J = 6.9 Hz), 6.68 (d, 1H, J = 2.8 Hz), 7.45 (d, 1H, J = 2.8) ppm.	(In CDCl ₃): 26.4, 35.3, 38.9, 113.4, 119.4, 164.7, 171.0 ppm.
18	(In CDCl ₃) 2.50 (m, 2H), 2.69 (m, 2H), 3.37 (s, 6H), 3.40 (t, 2H, J = 5.4 Hz); 4.38 (t, 1H, J = 5.4); 6.21 (s, 1H) ppm.	(In CDCl ₃) 28.3, 29.5, 41.0, 54.4, 102.4, 172.3, 176.7 ppm.
17	(In CDCl ₃) 2.52 (t, 2H, J = 7.0 Hz), 2.73 (s, 4H), 2.87 (t, 2H, J = 7.0 Hz); 3.28 (s, 6H), 3.30 (m, 2H); 4.38 (t, 1H, J = 5.0 Hz); 6.21 (s, 1H) ppm.	(In CDCl ₃) 28.3, 29.5, 41.0, 54.4, 102.4, 172.3, 176.7 ppm.

Table S-2. ¹H-NMR Assignments for PAS Polymers

δ (ppm) (CDCl ₃)				
¹ H	PAS-7600	PAS-5600	PAS-4000	PAS-2800
CONH	7.57, 7.14	7.57, 7.14 (t, J=5 Hz)	7.59, 7.14	7.61, 7.15 (t, J=5 Hz)
e, f	4.04	4.04, 4.02	4.04	4.04, 4.03
c, d	3.61- 3.54	3.62 - 3.52	3.59	3.62- 3.52
a, p	3.36- 3.29	3.38-3.28	3.35- 3.29	3.36-3.28
s/t	2.62 (poorly defined t), 2.49, poorly defined t)	2.62 (t, J=6.5 Hz), 2.49 (t, J=6.5 Hz)	2.49	2.62 (t, J=6.5 Hz), 2.49 (t, J=6.5 Hz)
b	1.81 (m)	1.78 (m)	1.78 (m)	1.78 (m)
q	1.55	1.55	1.55	1.55

Table S-3. ^{13}C -NMR Assignments for PAS Polymers

δ (ppm) (CDCl_3)				
^{13}C	PAS-7600	PAS-5600	PAS-4000	PAS-2800
-CO-	174.3, 172.0, 168.9, 168.6	174.4, 172.1, 168.9, 168.7	174.4, 172.1, 169.0, 168.7	174.3, 172.1, 169.0, 168.8
e, f	70.8	70.8	70.8	70.7
c, d	70.0, 69.8, 69.3	70.0, 69.8, 69.3	70.0, 69.8, 69.7, 69.3, 69.2	70.1, 70.0, 69.8, 69.3, 69.1
a, p	38.4, 37.8, 37.0	38.4, 37.8, 37.0	38.5, 37.8, 37.0	38.5, 37.8, 37.0, 36.9
s/t	29.5	30.7	37.0	30.7
b	26.6	26.6	26.6	26.6
s/t	28.6	28.6	28.6	29.0
q	29.0	29.0	29.0	29.0

Table S-4. ^1H -NMR Assignments for PATSS Polymers

δ (ppm) (CDCl_3)			
^1H	PATSS-5700	PATSS-5200	PATSS-2800
CONH	6.89	6.89	6.96, 6.85, 6.79
c, d	3.61-3.48	3.58-3.47	3.57, -3.47
a	3.27 (q, J=6.3 Hz)	3.26 (q, J=6.2 Hz)	3.28 (q, J=5.8 Hz)
s/t	2.93 (t, J=7.2 Hz)	2.91 (t, J=7.2 Hz)	2.91 (t, J=7.2 Hz)
r	2.80	2.78	2.78
g	2.75 (t, J=7.2 Hz)	2.74 (t, J=7.2 Hz)	2.74 (t, J=6.7 Hz)
s/t	2.5 4(t, J=7.2 Hz)	2.52 (t, J=7.2 Hz)	2.52 (t, J=7.2 Hz)
h	2.41 (t, J=7.2 Hz)	2.39(t, J=7.2 Hz)	2.39(t, J=7.2 Hz)
b	1.73 (q, J=6.3 Hz)	1.71 (q, J=6.2 Hz)	1.71 (q, J=5.8 Hz)

Table S-5. ^{13}C -NMR Assignments for PATSS Polymers

^{13}C	δ (ppm) (CDCl_3)		
	PATSS-5700	PATSS-5200	PATSS-2800
-CO-	172.3, 171.3, 169.8, 169.1, 168.3	171.2, 169.8, 169.0, 168.2	171.3, 171.1, 169.8, 168.2
c, d	70.4, 70.0, 69.6	70.3, 69.9, 69.8, 69.7, 69.5	70.3, 70.1, 69.9, 69.8, 69.7, 69.7, 69.6, 69.5
a	38.0, 37.6	37.9, 37.6, 37.5	37.6, 37.5
g	36.5	36.4	36.5
s/t	30.4	30.3	30.4
b	30.0, 28.8	28.9, 28.8	28.9, 28.8, 28.0
h	27.7	27.7, 27.6	27.7
s/t	26.7	26.7	26.7
r	25.5	25.5	25.5

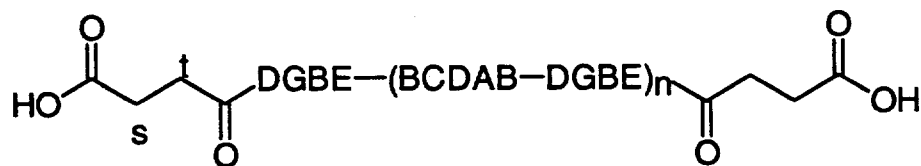
Table S-6. ^1H -NMR Assignments for PADSC and PADA Polymers

δ (ppm) (CDCl_3)			
^1H	PADCS-3000	PADA-2800 Bis(Acetal)	PADA-2800
CHO	NA	NA	9.56
CONH	6.90, 7.33, 7.50	6.70, 6.90, 7.51	7.58
$(\text{MeO})_2\text{CH}$	NA	4.30 (t, $J = 4.8$ Hz)	NA
e, f	4.03	3.96	4.02
c, d	3.52- 3.58	3.45- 3.54	3.54- 3.66 (m)
a, p, u	3.27- 3.35 (m)	3.23- 3.29 (m)	3.27-3.35 (m)
r	2.80	NA	NA
s/t	NA	2.44	2.50
b	1.74, 1.76, 1.78	1.71 (m)	1.76 (m)
q	1.53	1.48	1.53
MeO-	NA	3.29	NA

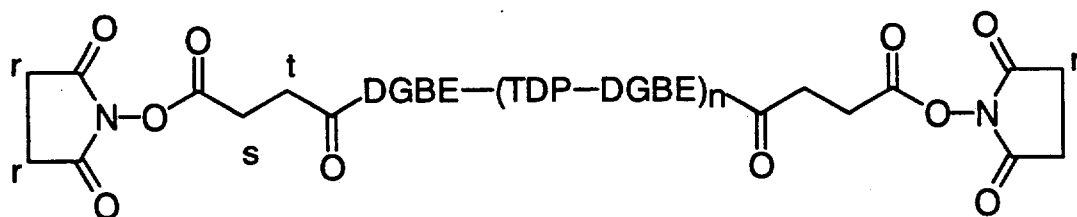
Table S-7. ^{13}C -NMR Assignments for PADSC and PADA Polymers

δ (ppm) (CDCl_3)			
^{13}C	PADSC-3000	PADAS-2800	PADA-2800
CHO	NA	NA	195.5
-CO-	172.5, 172.1, 170.2, 169.1, 168.8, 168.7	172.5, 172.1, 170.2, 169.0, 168.8, 168.7	172.2, 169.2, 168.9
CO (urethane)	151.6	NA	NA
v	NA	102.3	100.5
c, d, e, f	70.9, 70.2, 70.1, 70.0, 69.9, 69.7, 69.4, 69.3	70.9, 70.2, 70.1, 69.9, 69.7, 69.4, 69.3	70.9, 70.3, 70.2, 69.9, 69.5
u	NA	40.8	50.1
a, p	38.5, 37.3, 37.2	38.5, 37.7, 37.1, 37.0	38.6, 37.9, 37.2
q	29.1, 29.0	29.0	29.1
s/t	NA	31.4	31.5
b	26.7	26.6	26.7
s/t	NA	28.8	26.7
r	25.5	NA	NA

Figure 1-S



PAS



PATSS

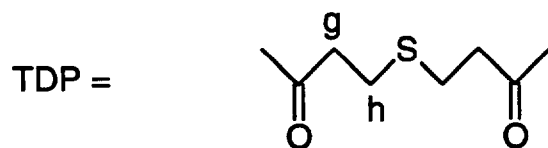
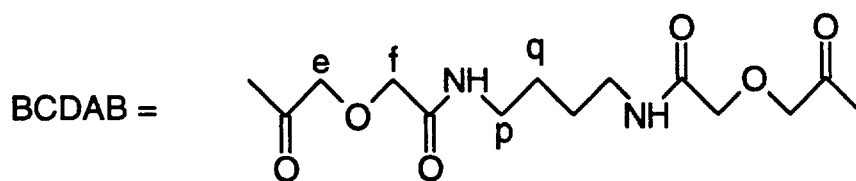
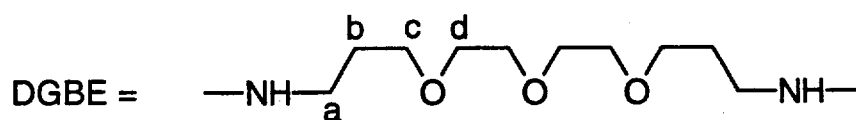


Figure 2-S

