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The Synthesis of Amphiphilic HT Alternating Copolymers of Thiophenes.

Synthesis of Molecules and Polymers in Scheme 1.

Molecule **1** is prepared from 2-bromo-3-bromomethylthiophene by a nucleophilic displacement with the 2-lithio salt of 2,4,4-trimethyloxazoline.

2-bromo-3-bromomethylthiophene.

CAUTION: COMPOUND IS A LACRYAMATOR, PHOTO TOXIN, AND STRONG ALKYLATING AGENT. 2-bromo-3-methylthiophene (17.8g, 100mmol) and benzene (150 ml) were placed round bottomed flask. This mixture was heated to reflux. N-bromosuccinimide (178g, 100mmol) and AIBN (1g) was added and the heat was removed. Once the exotherm subsided, the mixture was refluxed for an hour and cooled to room temperature. The solvent was removed *in vacuo* and hexanes was added. The solution was then filtered through a plug of silica gel to remove the succinimide. The filtrate was concentrated *in vacuo* and the product purified by distillation (74°C @ ~0.1 mm of Hg) to yield 17.7g (69% yield) of 2-bromo-3-bromomethylthiophene. Purity of compound is >99% by GC/MS. ^1H NMR 7.26(d, 1H), 7.0(d, 1H), 4.46(s, 2H).

2-Bromo-3-(2(4,4-dimethyloxazolin-2-yl) ethyl)thiophene (1).

2,4,4-Trimethyloxazoline (6.2g, 55mmol) and freshly distilled dry THF (150ml) were placed in a round bottomed flask under nitrogen. The mixture was cooled to -78°C and 22 ml of 2.5M solution of BuLi in hexanes (55mmol) was added dropwise. After 30 min, 2-bromo-3-bromomethylthiophene (13g, 50mmol) was added at -78°C. The cooling bath was removed and the mixture was stirred for an hour. The solution was then extracted with ether. The ethereal solution was dried over MgSO_4 and solvent removed *in vacuo*. Unreacted starting material was removed by filtration through a column of silica gel with hexanes and the solvent removed *in vacuo*. The product was purified by distillation under vacuum (100°C @ 0.1 mm of Hg) to yield 8.9g (62% yield) of 2-Bromo-3-(2(4,4-dimethyloxazolin-2-yl) ethyl)thiophene. Purity of compound is >99% by GC/MS. ^1H NMR 7.19(d, 1H), 6.84(d, 1H), 3.91(s, 2H), 2.89(t, 2H), 2.52(t, 2H), 1.24(s, 6H).

2-Trimethylstanny-4-dodecylthiophene (2).

3-dodecylthiophene (40 mmol), TMEDA (44 mmol) and 100 ml dry ether is placed in a 250 rbf under Ar and cooled on an ice/salt bath. 16 ml 2.5 M BuLi/hexanes (40 mmol) is added dropwise after which the cooling is removed and the mixture is refluxed for 1 hr. White suspension is cooled again on ice/salt followed by addition of 45 ml 1.0 M Me_3SnCl in THF (45 mmol) over 10 min. Ice/salt bath removed after 45 min. and mixture stirred at RT overnight under Ar. White suspension is hydrolyzed with 50 ml NH_4Cl (aq). Separated, waterphase washed once with 50 ml ether. Combined org. phase washed once with water and once with brine. Dried over MgSO_4 , filtered and evap-Leaving clear yellow oil.

Kugel-rohr distillation : yields pure fractions at 180-190°/0.05 mmHg. 17.45 g clear pale yellow liquid. Yield : 34.9 mmol = 87 %. ^1H NMR 7.41(d, 1H, $J=2$ Hz), 6.98(d, 1H, $J=2$ Hz), 2.65(t, 2H), 1.7-1.55(m, 2H), 1.35-1.15(m, 18H), 0.85(t, 3H), 0.22(s, 9H).

3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl)ethyl)-5,2'-bithiophene .

2-Bromo-3-(2(4,4-dimethyloxazolin-2-yl)ethyl)thiophene (4.6g, 17mmol) was mixed along with Pd₂(dba)₃ (0.7g, 5 mol%), PPh₃ (0.9g, 20 mol%), CuO (1.4g, 17mmol) and DMF (100ml) in RB flask under nitrogen and stirred for 10 min. To this 3-dodecyl-5-trimethylstannylthiophene (10.4g, 25mmol) was added and the mixture was stirred at 110°C for 12 hrs. It was then allowed to cool down to RT, filtered and filtrate poured into ethyl ether. The resulting solution was washed with water to remove DMF. The organic layer was then dried over anhy. MgSO₄ and solvent removed *in vacuo*. The product was purified by flash chromatography over silica gel with 70:30 hexanes, ethyl acetate as eluent to yield 3.7g (57% yield) of 3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl)ethyl)-2',5-bithiophene. 7.14(d, 1H), 6.95(br. s, 1H), 6.92(d, 1H), 6.88(br. s, 1H), 3.85(s, 2H), 3.1(t, 2H), 2.55(m, 4H), 1.65-1.55(m, 2H), 1.4-1.2(m, 24H), 0.85(t, 3H).

2-Bromo-3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl)ethyl)-5,2'-bithiophene. NMR enclosed.

To 6.5g of 3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl)ethyl)-5,2'-bithiophene (14 mmol) in THF (25 ml), NBS (2.5g, 14 mmol) was added and the mixture was allowed to stir for 45 min to an hour. The solvent was then removed *in vacuo* and hexane was added to the concentrate. The solid succinimide was removed by filtration and the solvent was removed *in vacuo*. The product was purified by column chromatography to yield 5.2g (70% yield) of 2-bromo-3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl)ethyl)-5,2'-bithiophene. ¹H NMR 7.17(d, 1H), 6.94(d, 1H), 6.8(d, 1H), 3.9(s, 2H), 3.05(t, 2H), 2.55(m, 4H), 1.65-1.55(m, 2H), 1.4-1.2(m, 24H), 0.85(t, 3H). ¹³C NMR 164.66, 142.49, 137.54, 135.23, 130.93, 129.58, 127.14, 124.21, 108.76, 78.99, 66.93, 31.86, 29.59, 29.53, 29.50, 29.34, 29.27, 29.19, 28.97, 28.3, 25.4, 22.60, 13.99. Elemental analysis C 60.62% (calculated 60.22%), H 7.63% (calculated 7.43%).

2-bromo-3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl) ethyl)-5'-trimethylstannyl-2',5-bithiophene (3). NMR enclosed.

Dry diisopropylamine (0.7ml, 5 mmol) and freshly distilled, dry THF (20 ml) were placed in a round bottomed flask. This mix was cooled to -78°C and 2ml of 2.5M BuLi in hexane was added. The solution was warmed to 0°C, stirred at that temperature for 5 min and cooled back to -78°C. To this reaction mixture containing LDA, a solution of 2-bromo-3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl) ethyl)-2',5-bithiophene (2.7g, 5 mmol) in 5ml of dry THF was added slowly and the solution was stirred at -78°C for an hour. To this mixture 5 ml of 1M Me₃SnCl solution in hexanes (5 mmol) was added at -78°C and allowed to warm up to RT slowly. The reaction was worked up by pouring into water and extracting with ethyl ether. The organic layer was dried over anhy. MgSO₄ and solvent removed *in vacuo*. The product was purified by flash chromatography to yield 2.2g (61% yield) of 2-bromo-3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl) ethyl)-5'-trimethylstannyl-2',5-bithiophene. ¹H NMR 7.0(s, 1H), 6.79(s, 1H), 3.15(t, 2H), 2.6(m, 4H), 1.8-1.65(m, 2H), 1.35-1.15(m, 24H), 0.85(t, 3H), 0.35(s, 9H).

Polymerization of 2-bromo-3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl)ethyl)-5'-trimethylstannyl-2',5-bithiophene (4). NMR enclosed.

2-bromo-3-dodecyl-3'-(2,5,8-trioxanonyl)-5'-trimethylstannyl-2',5-bithiophene (3.5g, 5mmol) was mixed along with Pd₂(dba)₃ (0.228g, 5 mol%), PPh₃ (0.267g, 20 mol%), CuO (0.4g, 5mmol) and DMF (100ml) in RB flask under argon and the mixture was stirred at 110°C for 16 hrs. It was then allowed to cool down to RT and the polymer precipitated by pouring into methanol. The polymer was filtered, washed with methanol, water and methanol again. The residue was the Soxhlet extracted with methanol, hexanes and chloroform. Upon removal of solvent, the chloroform fraction yielded 1.5g (65% yield) of poly(3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl)ethyl)-2',5-bithiophene). ¹H NMR 7.15(s, 1H), 6.98(s, 1H), 3.9(s, 2H), 3.2(t, 2H), 2.8(m, 4H), 1.8-1.65(m, 2H), 1.35-1.15(m, 24H), 0.85(t, 3H).

Preparation of Poly(3-dodecyl-3'-(butylpropionyl)-2',5-bithiophene) (5a). NMR enclosed.

1g of poly(3-dodecyl-3'-(2(4,4-dimethyloxazolin-2-yl)ethyl)-2',5-bithiophene) was placed in a round bottomed flask along with 45 ml of n-butanol, 3ml of distilled water and 2 ml of conc.H₂SO₄. This suspension was stirred at 100-110°C for approximately 12 hours. The mixture was allowed to cool down to RT and poured into methanol to precipitate the polymer. The precipitate was isolated by filtration and washed with methanol, water and methanol again. Then the residue was Soxhlet extracted with methanol, hexanes and chloroform. Upon removal of the solvent *in vacuo* the chloroform fraction yielded 0.85g (84% yield) of poly(3-dodecyl-3'-(butylpropionyl)-2',5-bithiophene). ¹H NMR 7.0(br. s, 1H), 4.14(t, 2H), 3.15(t, 2H), 2.75(m, 4H), 1.8-1.65(m, 4H), 1.35-1.15(m, 20H), 0.95(t, 3H), 0.85(t, 3H).

Preparation of Poly(3-dodecyl-3'-(isopropionyl)-2',5-bithiophene) (5b). NMR enclosed. Procedure the same except isopropanol used.

Preparation of poly(3-dodecyl-3'-(tetramethylammonium propionate)-5,2'-bithiophene) (6). NMR enclosed.

Poly(3-dodecyl-3'-(butylpropionyl)-5,2'-bithiophene) (0.5 g) was dissolved in 100 ml of THF. To this solution 100 ml of methanol and 10 g of Me₃NOH.5H₂O were added and this mixture was refluxed for approximately 12 hours. The solvent was then removed *in vacuo* and 50 ml of 2M HCl was added to precipitate the insoluble propionic acid. The solution was filtered and the residue was washed with water and then by methanol. The polymer was then dried under vacuum to yield 400 mg of the propionic acid. The acid was suspended in 25 ml of methanol and an equivalent amount of Me₃NOH.5H₂O was added. This mixture was sonicated for 30 min. at approximately 50°C, after which the solvent was removed *in vacuo* to yield 0.47g of poly(3-dodecyl-3'-(tetramethylammonium propionate)-5,2'-bithiophene). ¹H NMR 7.1(br. s, 1H), 7.05(br. s, 1H), 4.9(s, 12H), 2.77(t, 2H), 2.49(t, 2H), 1.65(m, 2H), 1.555-1.2(m, 18H), 0.85(t, 3H)

2-Bromo-3-(2,5,8-trioxanonyl)thiophene (8).

Sodium hydride (3.6g, 150mmol) and THF (100ml) were placed in a round bottomed flask under nitrogen. Di(ethyleneglycol)methyl ether (18g, 150mmol) was added dropwise and the mixture was stirred for 15 min. 2-bromo-3-bromomethylthiophene (17.8g, 100mmol) was added and the reaction was stirred for one hour. The solution was poured into water and extracted with ether. The ethereal solution was dried over MgSO_4 and the solvent removed *in vacuo*. The product was purified by distillation (110°C @ 0.1 mm of Hg) to yield 25g (86% yield) of 2-Bromo-3-(2(4,4-dimethyloxazolin-2-yl)ethyl)thiophene. ^1H NMR 7.23(d, 1H), 7.0(d, 1H), 4.52(s, 2H), 3.66(m, 6H), 3.57(m, 2H), 3.39(s, 3H).

2-TMS-3-dodecyl-3'-(2,5,8-trioxanonyl)-2',5-bithiophene.

Tetrahydrofuran (100 ml) and 2-TMS-3-dodecylthiophene (8.2g, 25 mmol) were placed in a round bottomed flask. This mixture was cooled to -78°C and of 10 ml of 2.5M BuLi in hexanes (25 mmol) was added dropwise. The solution was stirred at -78°C for 45 min. This mixture was warmed to -60°C, and $\text{MgBr}_2\text{-Et}_2\text{O}$ (6.5g, 25 mmol) was added. The reaction was then slowly allowed to warm up to 0°C over and hour, whereupon all $\text{MgBr}_2\text{-Et}_2\text{O}$ had reacted. 2-bromo-3-(2,5,8-trioxanonyl)thiophene (**8**) (5.8g, 20 mmol) followed by 5 mol% of $\text{Ni}(\text{dppp})\text{Cl}_2$ (0.813g) was then added. The cooling bath was removed and the mixture was stirred overnight. The mixture was then poured onto a cold 2N HCl followed by extraction with diethyl ether. The organic layers were combined and dried over MgSO_4 and solvent removed *in vacuo*. The product was purified by flash chromatography using 75:25 hexanes, ethyl acetate as eluent to yield 8.4g (78% yield) of the title compound. ^1H NMR 7.15(d, 1H), 7.1(d, 1H), 7.08(s, 1H), 4.65(s, 2H), 3.66(m, 6H), 3.57(m, 2H), 3.39(s, 3H), 2.65(t, 2H), 1.7-1.55(m, 2H), 1.35-1.15(m, 18H), 0.85(t, 3H), 0.32(s, 9H).

Preparation of 3-Dodecyl-3'-(2,5,8-trioxanonyl)-2',5-bithiophene.

Freshly distilled dry THF (100ml) and 1 mmol/g dispersion of Bu_4NF on silica gel (25g, 25mmol) were placed under nitrogen in a round bottomed flask. This mixture was stirred for 5 min at RT and 2-TMS-3-dodecyl-3'-(2,5,8-trioxanonyl)-5,2'-bithiophene (13.5g, 25mmol) was added and allowed to stir for 15 min at RT. Then the reaction mixture was filtered and the residue was washed with ether. The filtrate and ether washings were combined and washed with water. The organic layer was dried over anhy. MgSO_4 and solvent removed *in vacuo*. The product was purified by flash chromatography (75:25 hexanes, EtOAc) to yield 9.4g (80% yield) of 3-dodecyl-3'-(2,5,8-trioxanonyl)-2',5-bithiophene. ^1H NMR 7.15(d, 1H), 7.1(d, 1H), 6.98(br. s, 1H), 6.88(br. s, 1H), 4.55(s, 2H), 3.66(m, 6H), 3.57(m, 2H), 3.39(s, 3H), 2.65(t, 2H), 1.7-1.55(m, 2H), 1.35-1.15(m, 18H), 0.85(t, 3H).

Preparation of 2-Bromo-3-dodecyl-3'-(2,5,8-trioxanonyl)-2',5-bithiophene (9**). NMR enclosed.**

To 4.2g of 3-dodecyl-3'-(2,5,8-trioxanonyl)-5,2'-bithiophene (9 mmol) in THF (20 ml), NBS (1.7g, 9.5 mmol) was added and the mixture was allowed to stir for 45 min to an hour. The solvent was then removed *in vacuo* and hexane was added to the concentrate. The solid succinimide was removed by filtration and the solvent was removed *in vacuo*. The product was purified by column chromatography to yield 3.4g (70% yield) of 2-bromo-3-dodecyl-3'-(2,5,8-trioxanonyl)-2',5-bithiophene. ^1H NMR 7.17(d, 1H),

7.08(d, 1H), 6.84(s, 1H), 4.55(s, 2H), 3.66(m, 6H), 3.57(m, 2H), 3.39(s, 3H), 2.55(t, 2H), 1.7-1.55(m, 2H), 1.35-1.15(m, 18H), 0.85(t, 3H). ^{13}C NMR 142.61, 135.49, 134.73, 133.56, 130.02, 127.43, 123.98, 109.31, 71.94, 70.56, 70.50, 69.46, 66.69, 58.82, 31.81, 29.53, 29.49, 29.46, 29.39, 29.30, 29.21, 29.14, 22.55, 13.93. Elemental analysis C 57.55% (calculated 57.25%), H 7.51% (calculated 7.52%).

Preparation of Poly(3-dodecyl-3'-(2,5,8-trioxanonyl)-2',5-bithiophene) (10). NMR enclosed.

Kumada coupling: Dry diisopropylamine(0.7ml, 5 mmol) and freshly distilled, dry THF (20 ml) were placed in a round bottomed flask. This mix was cooled to -78°C and 2ml of 2.5M BuLi in hexane was added. The solution was warmed to 0°C, stirred at that temperature for 5 min and cooled back to -78°C. To this reaction mixture containing LDA, a solution of 2-bromo-3-dodecyl-3'-(2,5,8-trioxanonyl)-2',5-bithiophene (**6a**) (2.7g, 5 mmol) in 5ml of dry THF was added slowly and the solution was stirred at -78°C for an hour. This mixture was cooled to -60°C, MgBr₂·Et₂O (1.3g, 5 mmol) was added, and then the reaction was stirred at -60°C for 20 min, at -40°C for 15 min. The reaction was then slowly allowed to warm up to 0°C, whereupon all MgBr₂·Et₂O had reacted. To the above mixture 0.5 mol% (13.5 mg) of Ni(dppp)Cl₂ was added and the mixture was stirred at RT for 12-16 hrs. The polymer was then precipitated with MeOH and the resulting precipitate was then filtered and washed with MeOH, H₂O and MeOH again. The residue was then dried and purified by Soxhlet extraction with methanol, hexanes and, chloroform and carbon disulfide. Upon removal of solvent *in vacuo*, the chloroform fraction yielded 0.3g (13% yield)). ^1H NMR 7.17(s, 1H), 7.04(s, 1H), 4.65(s, 2H), 3.75-3.5(m, 6H), 3.4(s, 3H), 2.8(t, 2H), 1.8-1.65(m, 2H), 1.35-1.15(m, 18H), 0.85(t, 3H).

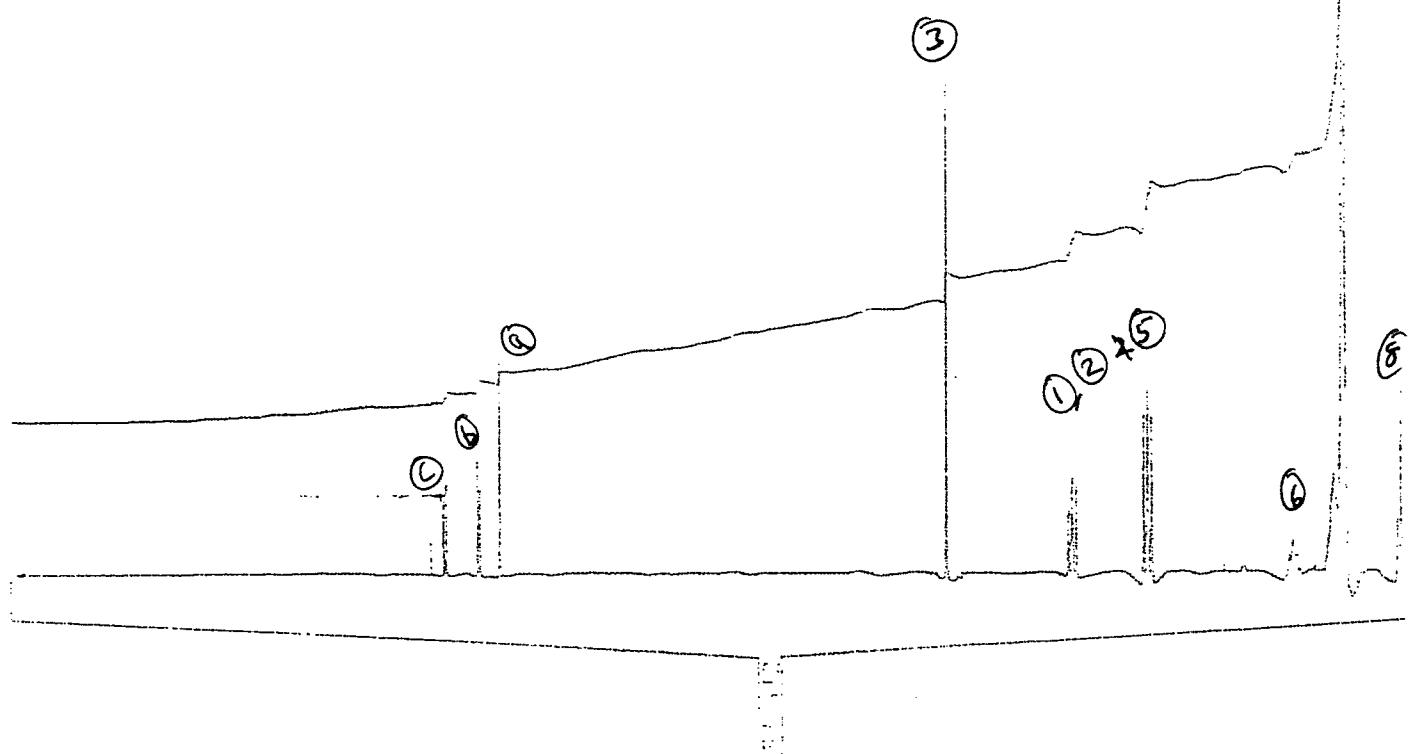
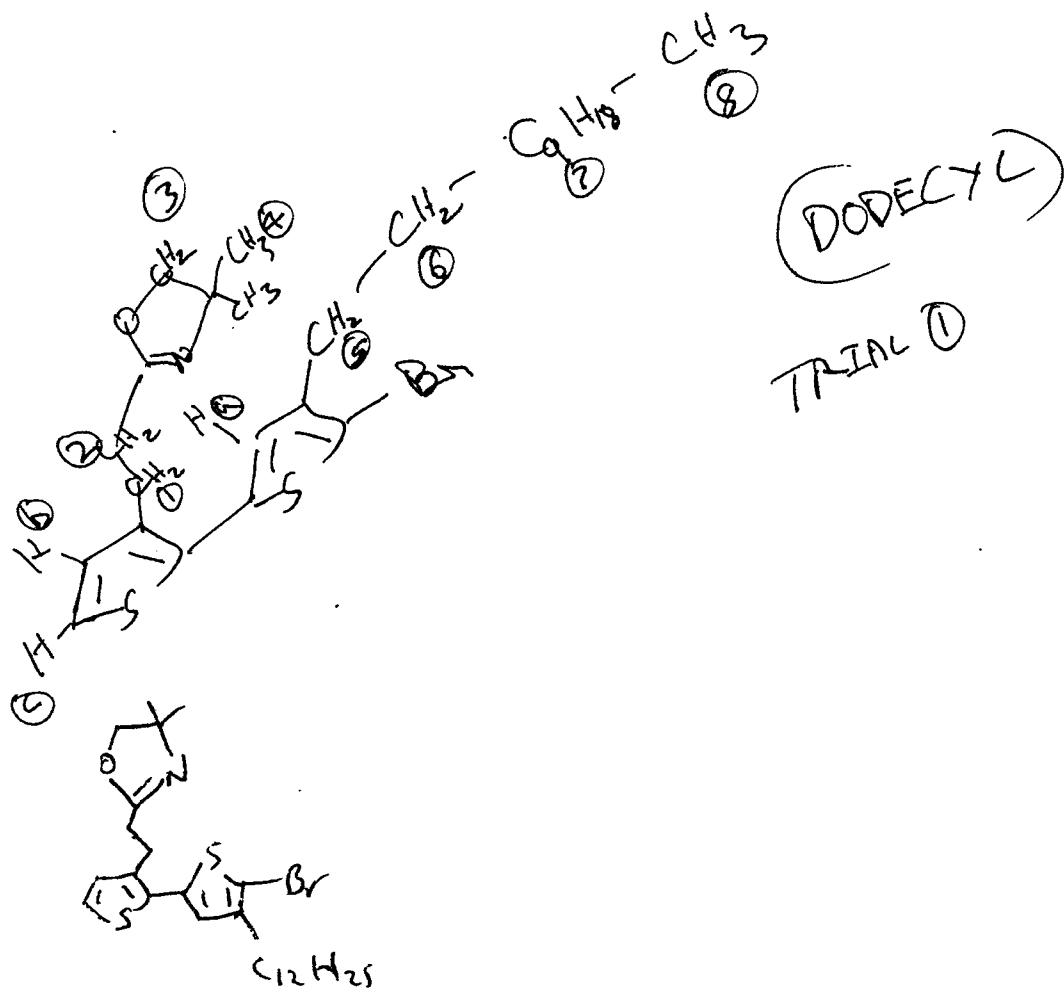
Stille coupling (not shown).

Preparation of 2-bromo-3-dodecyl-3'-(2,5,8-trioxanonyl)-5'-trimethylstannyl-2',5-bithiophene. Dry diisopropylamine (0.7ml, 5 mmol) and freshly distilled, dry THF (20 ml) were placed in a round bottomed flask. This mix was cooled to -78°C and 2ml of 2.5M BuLi in hexane was added. The solution was warmed to 0°C, stirred at that temperature for 5 min and cooled back to -78°C. To this reaction mixture containing LDA, a solution of 2-bromo-3-dodecyl-3'-(2,5,8-trioxanonyl)-5,2'-bithiophene (**9**) (2.7g, 5 mmol) in 5ml of dry THF was added slowly and the solution was stirred at -78°C for an hour. To this mixture 5 ml of 1M Me₃SnCl solution in hexanes (5mmol) was added at -78°C and allowed to warm upto RT slowly. The reaction was worked up by pouring into water and extraction with ethyl ether. The organic layer was dried over anhy.MgSO₄ and solvent removed *in vacuo*. The product was purified by flash chromatography to yield 2.2g (61% yield) of 2-bromo-3-dodecyl-3'-(2,5,8-trioxanonyl)-5'-trimethylstannyl-5,2'-bithiophene. ^1H NMR 7.17(s, 1H), 7.04(s, 1H), 4.65(s, 2H), 3.75-3.5(m, 6H), 3.4(s, 3H), 2.8(t, 2H), 1.8-1.65(m, 2H), 1.35-1.15(m, 18H), 0.85(t, 3H), 0.4(s, 9H).

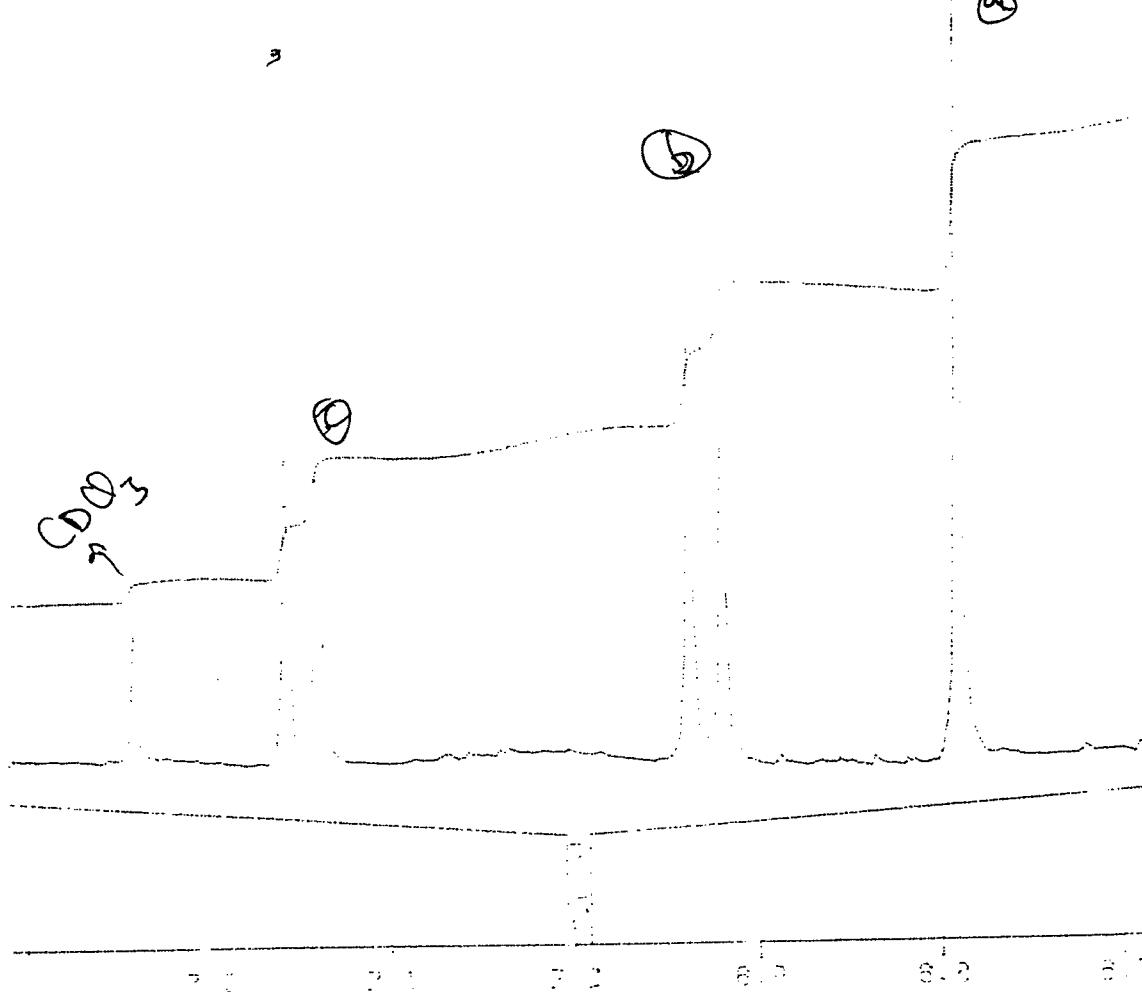
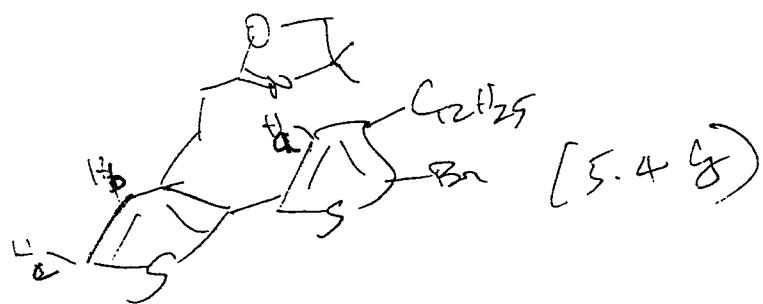
Not Shown. Polymerization of 2-bromo-3-dodecyl-3'-(2,5,8-trioxanonyl)-5'-trimethylstannyl-2',5-bithiophene. 2-bromo-3-dodecyl-3'-(2,5,8-trioxanonyl)-5'-trimethylstannyl-2',5-bithiophene (2.1g, 3mmol) was mixed along with Pd₂(dba)₃ (0.137g, 5 mol%), PPh₃ (0.16g, 20 mol%), CuO (0.24g, 3mmol) and DMF (100ml) in RB flask under argon and the mixture was stirred at 110°C for 16 hrs. It was then allowed to cool to RT and the polymer precipitated by pouring into methanol. The polymer was filtered, washed with methanol, water and methanol again. The residue was then Soxhlet

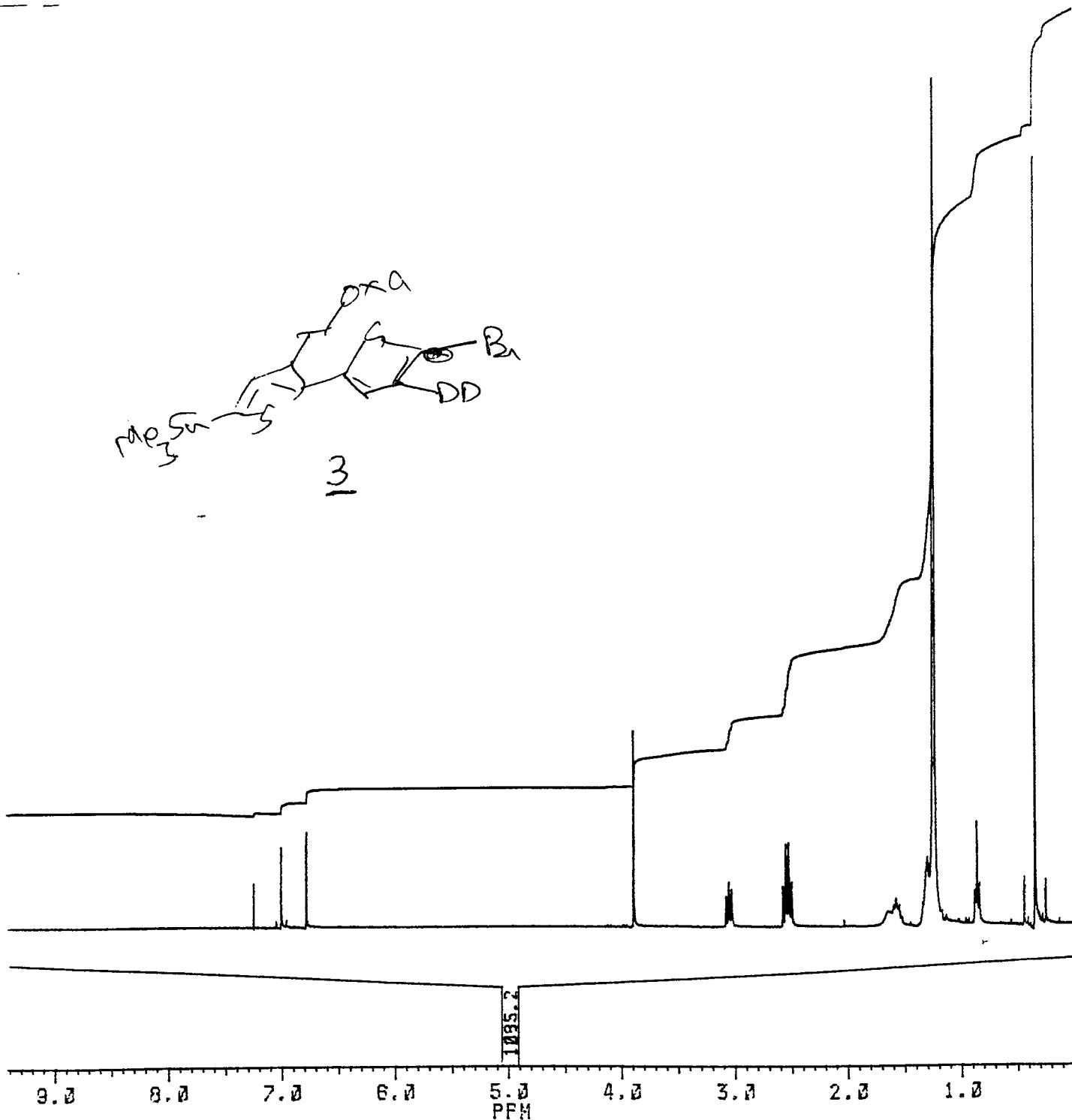
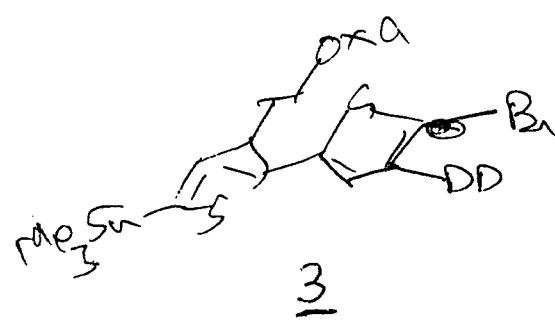
extracted with methanol, hexanes and chloroform. Upon removal of solvent, the chloroform fraction yielded 0.7g (50% yield) of poly(3-dodecyl-3'-(2,5,8-trioxanonyl)-5,2'-bithiophene). ^1H NMR 7.17(s, 1H), 7.04(s, 1H), 4.65(s, 2H), 3.75-3.5(m, 6H), 3.4(s, 3H), 2.8(t, 2H), 1.8-1.65(m, 2H), 1.35-1.15(m, 18H), 0.85(t, 3H).

Micro contact printing: The stamp used for microcontact printing resulted from curing of an elastomer (Sylgard 184, Dow Corning) over an VLSI integrated circuit kindly provided by H. Biebuyck. A 5 mM ethanolic (>99.8%, Fluka) solution of octadecylthiol (>99%, Fluka, recrystallized twice) "inked" the stamp surface. Brief contact between the inked stamp and a gold (200 Å) covered silicon wafer affected transfer of thiol molecules exclusively of points of intimate contact between the two. The remaining regions of the surface turned hydrophilic through submersion of the substrate in an 11 mM ethanolic solution of 11-mercaptopoundecanoic acid for 10 min. The resulting surface was flushed with ethanol and Milli-Q water before being placed in the the subphase of the trough.



(DODECYL)
TRIAL ①





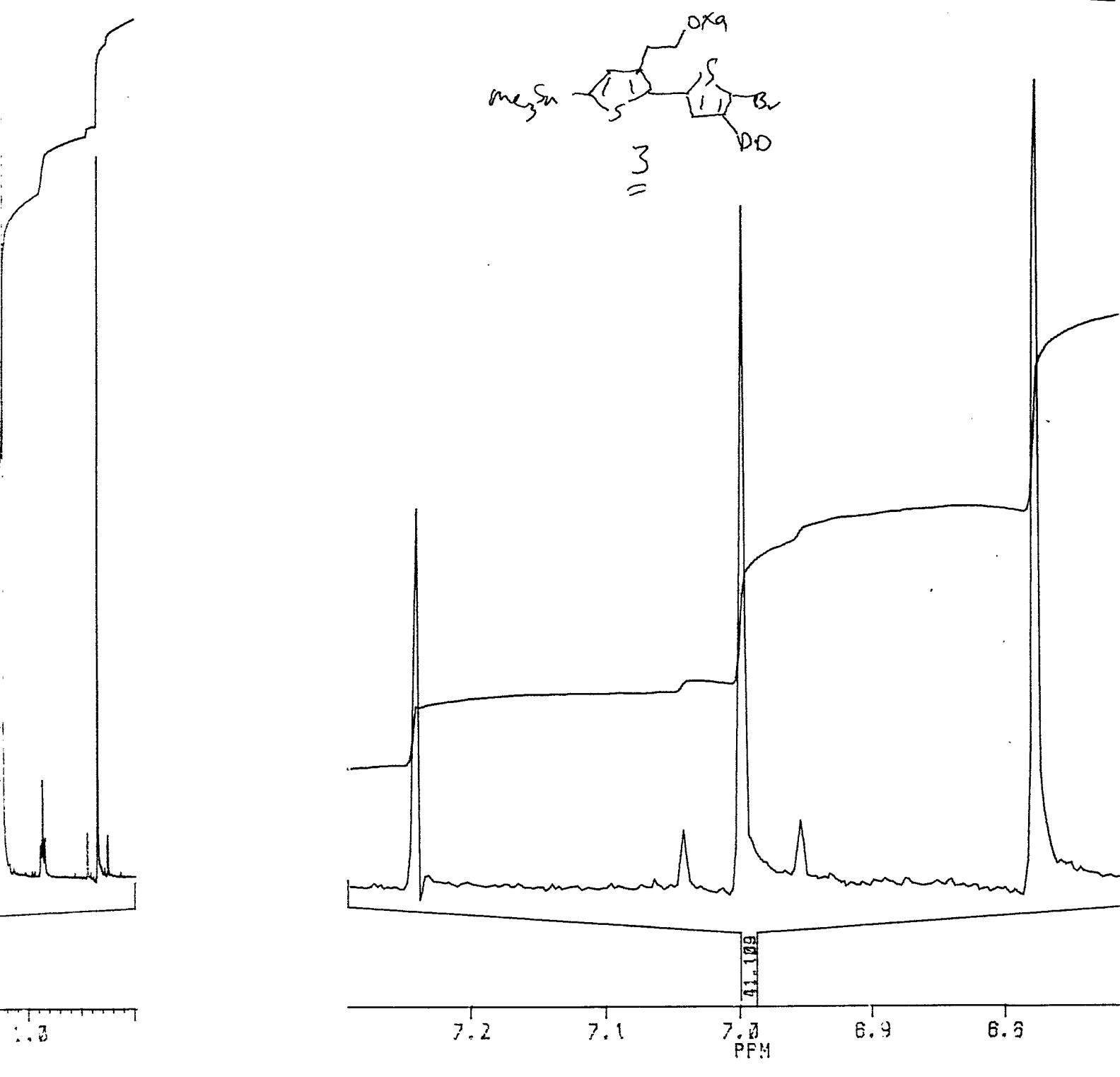
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Kemisk Institut
Københavns Universitet
Universitetsparken 5, DK 2100 København Ø. Tlf 35320308 Fax 31350609
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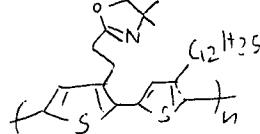
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Indleveret den 20/6-96 af D. Grønne / R. m (Cullough)

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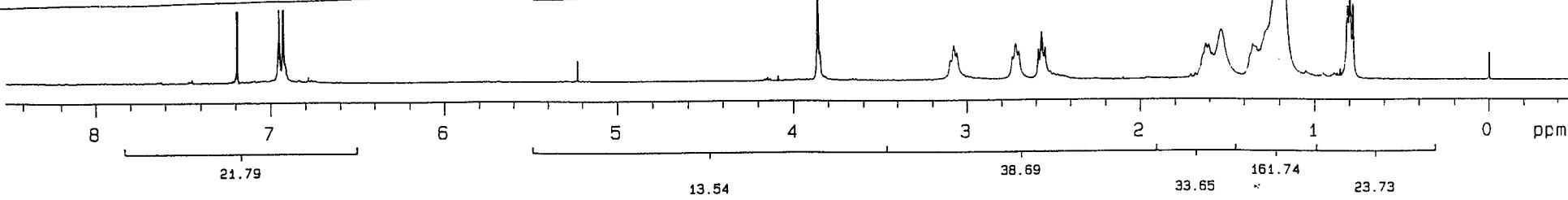
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Høster, fak!

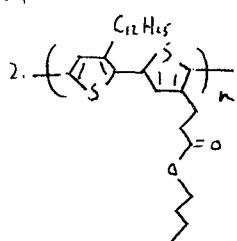
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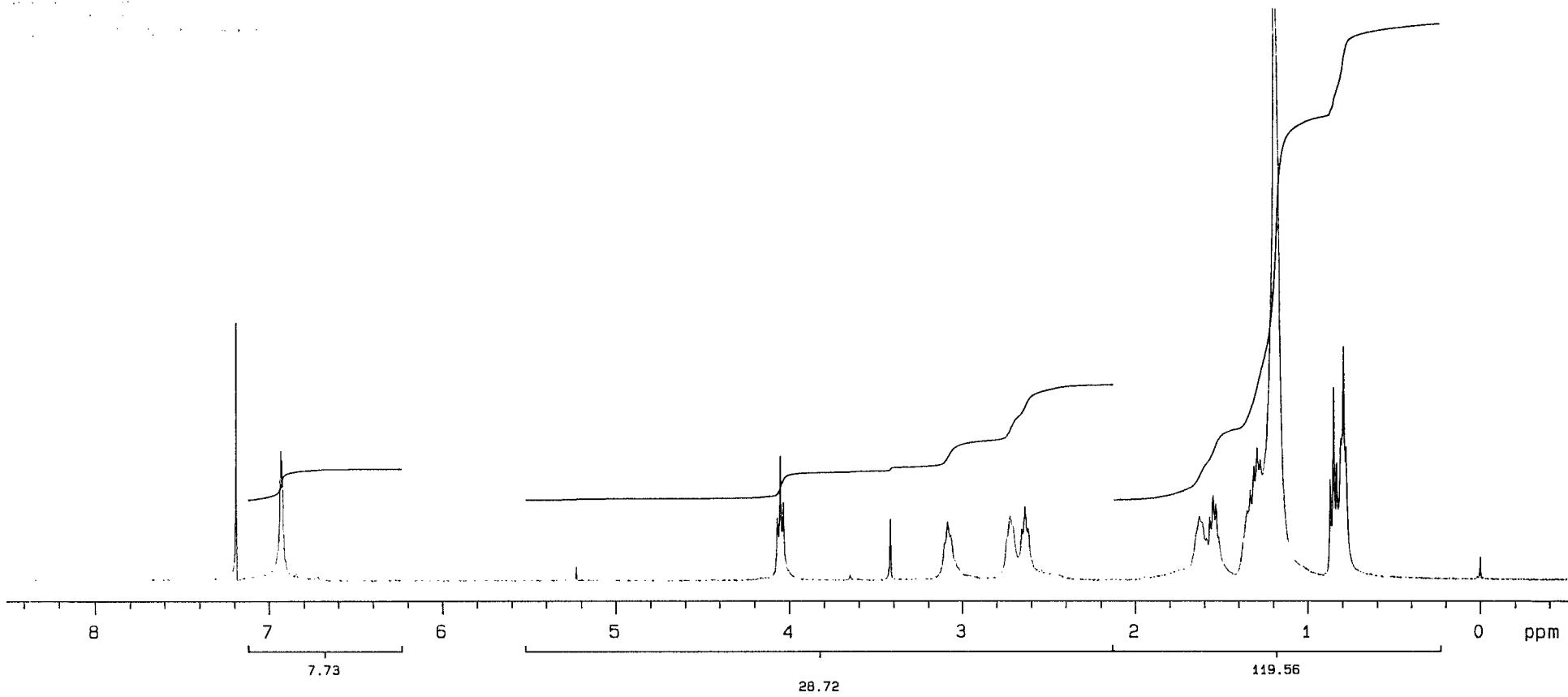
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5a

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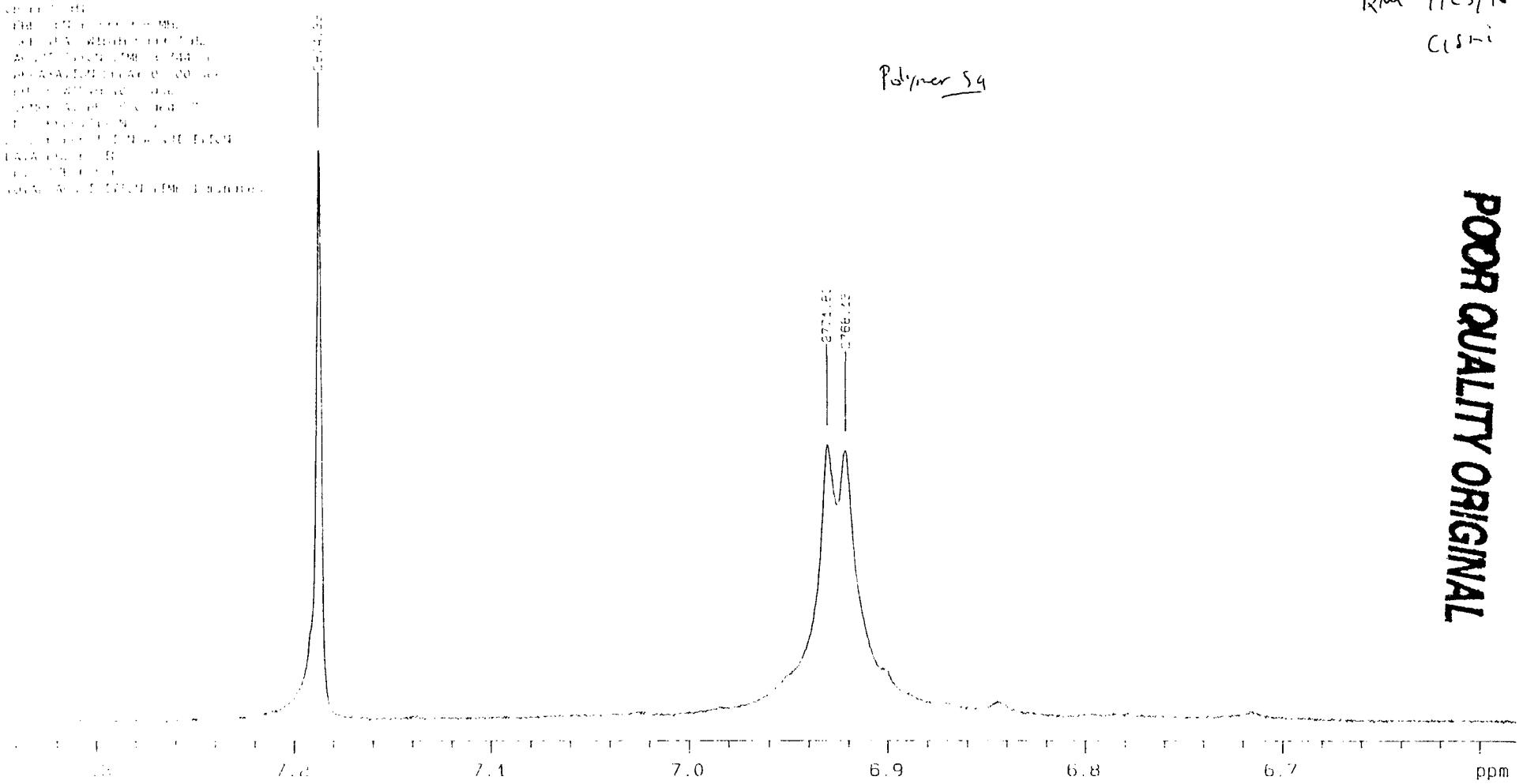
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MILK AND MILK PRODUCTS

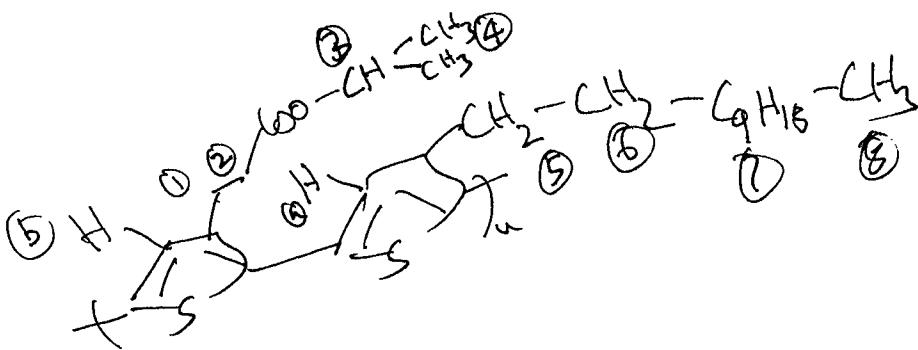
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C18-1

Polymer 59

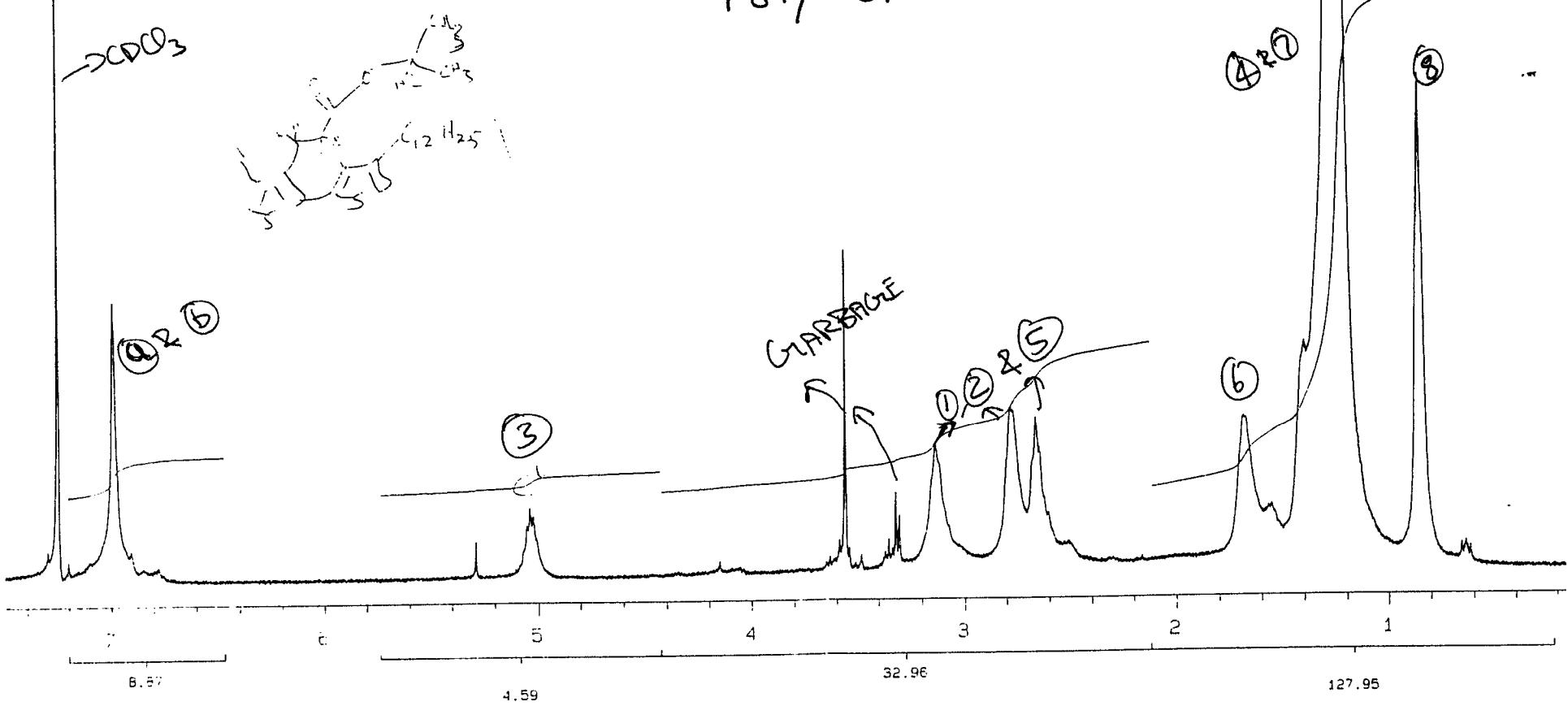


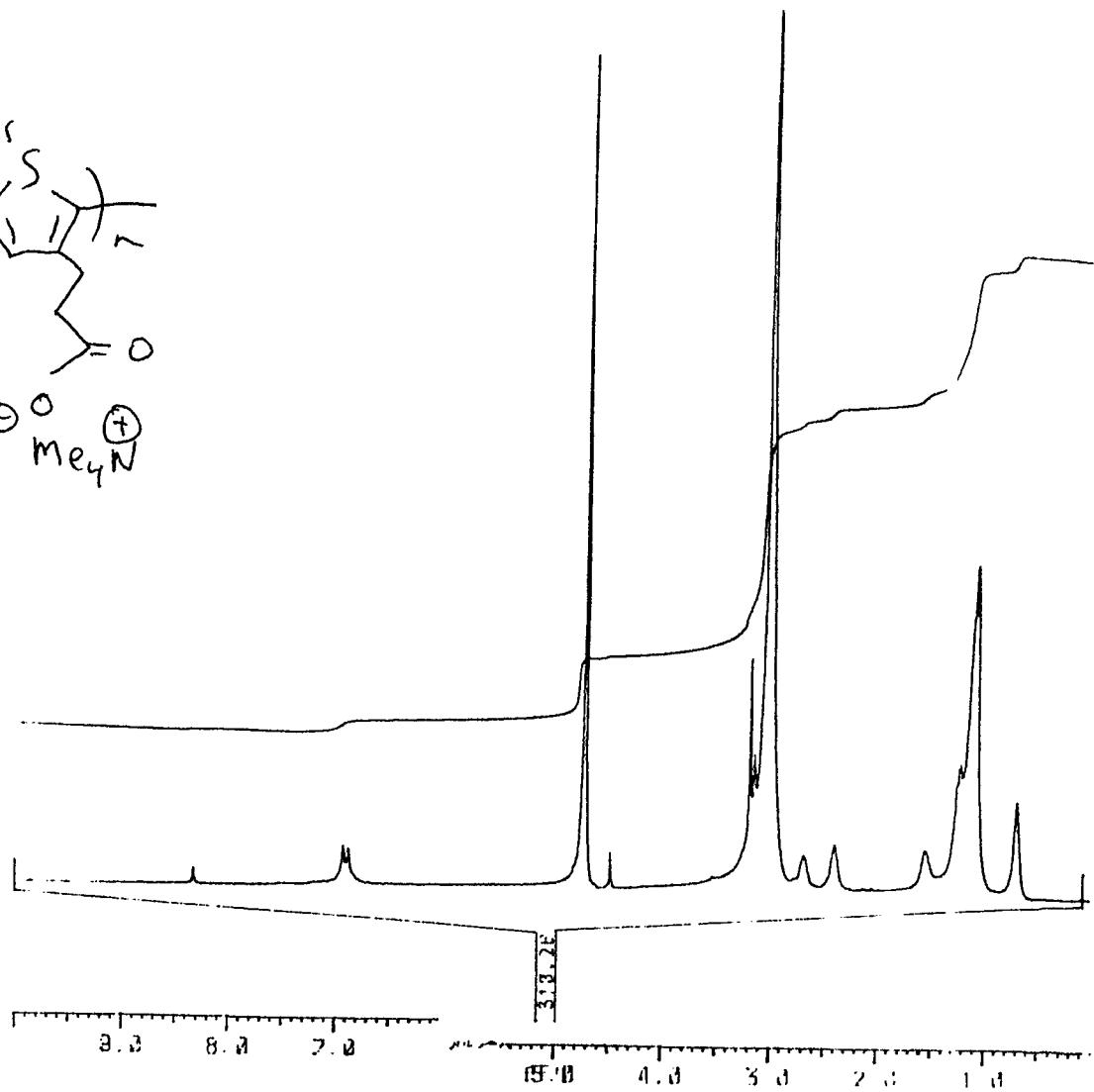
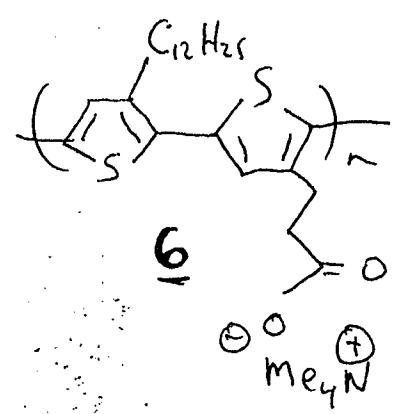
POOR QUALITY ORIGINAL

Mani-isoester
0845.fid
DATE: May 21 96
FILE: exp
SOLVENT: CDCl3
OBSERVE H1
FREQUENCY 399.938 MHz
SPECTRAL WIDTH 5999.7 Hz
ACQUISITION TIME 3.744 sec
RELAXATION DELAY 0.000 sec
PULSE WIDTH 10.0 usec
TEMPERATURE 25.0 deg. C.
NO. REPETITIONS 128
DOUBLE PRECISION ACQUISITION
DATA PHOCESSING
FT SIZE 65536
TOTAL ACQUISITION TIME 7 minutes



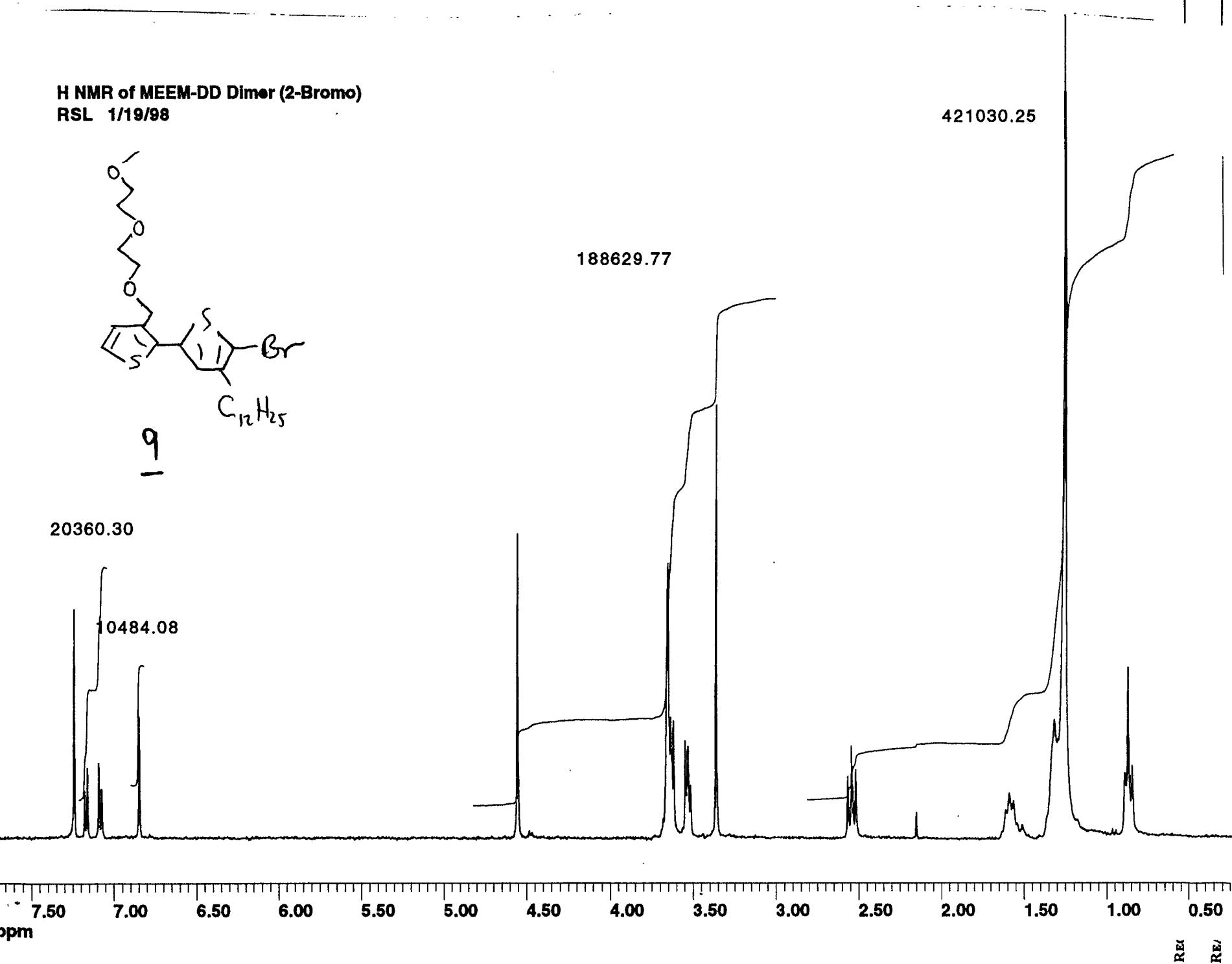
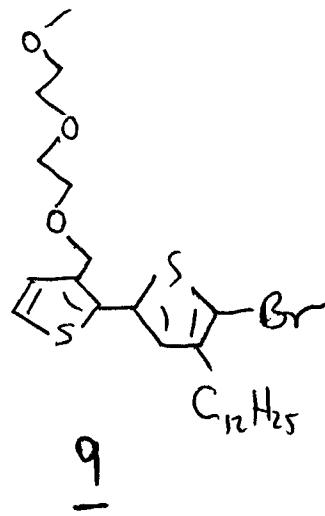
Polymer 5b



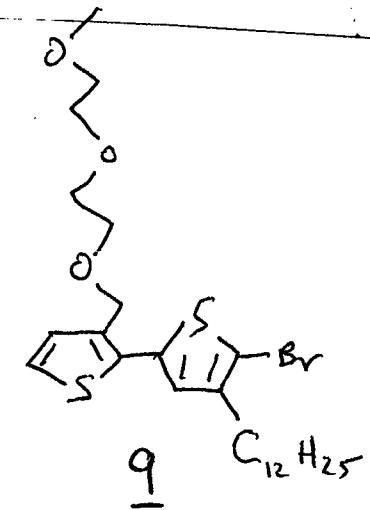


H NMR of MEEM-DD Dimer (2-Bromo)
RSL 1/19/98

421030.25

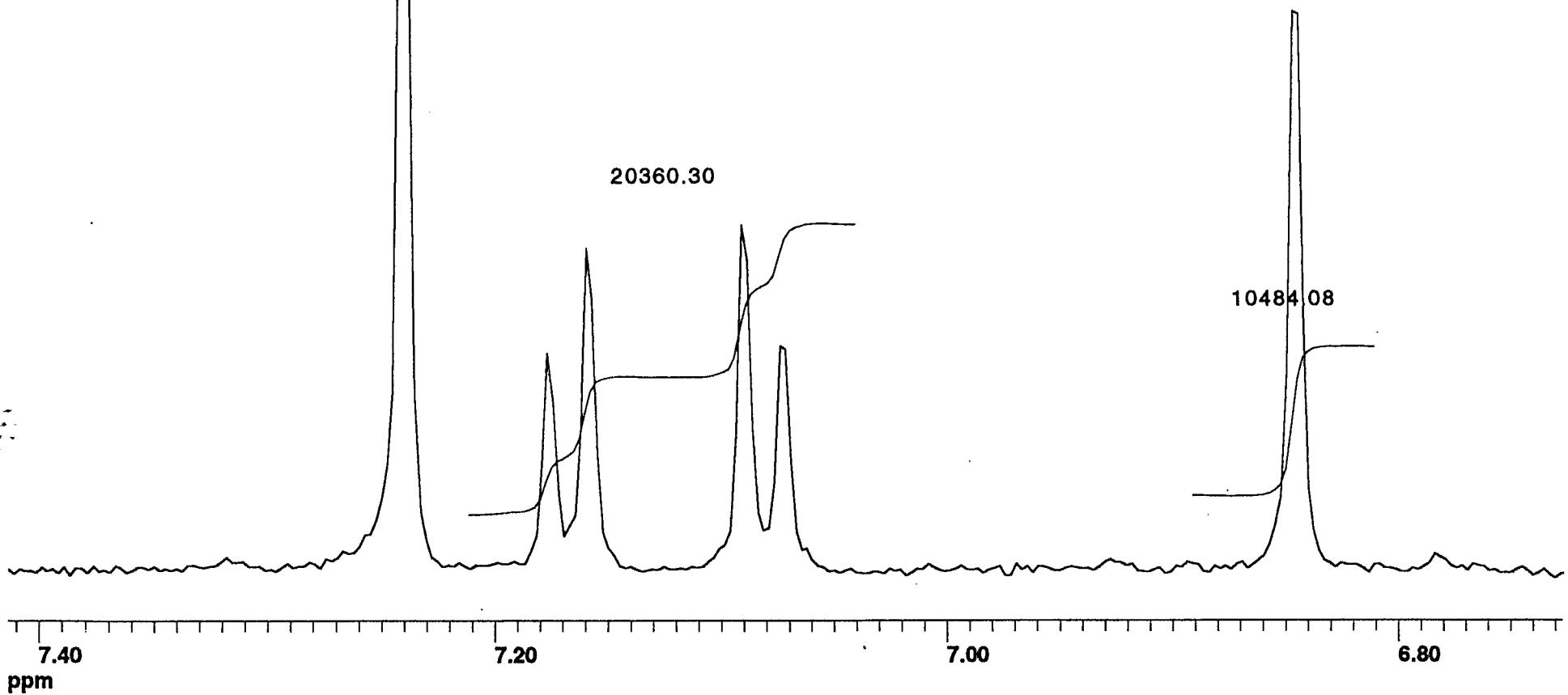


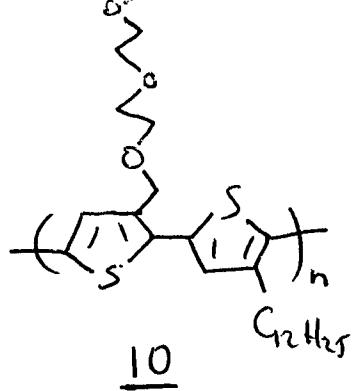
H NMR of MEEM-DD Dimer (2-Bromo)
RSL 1/19/98



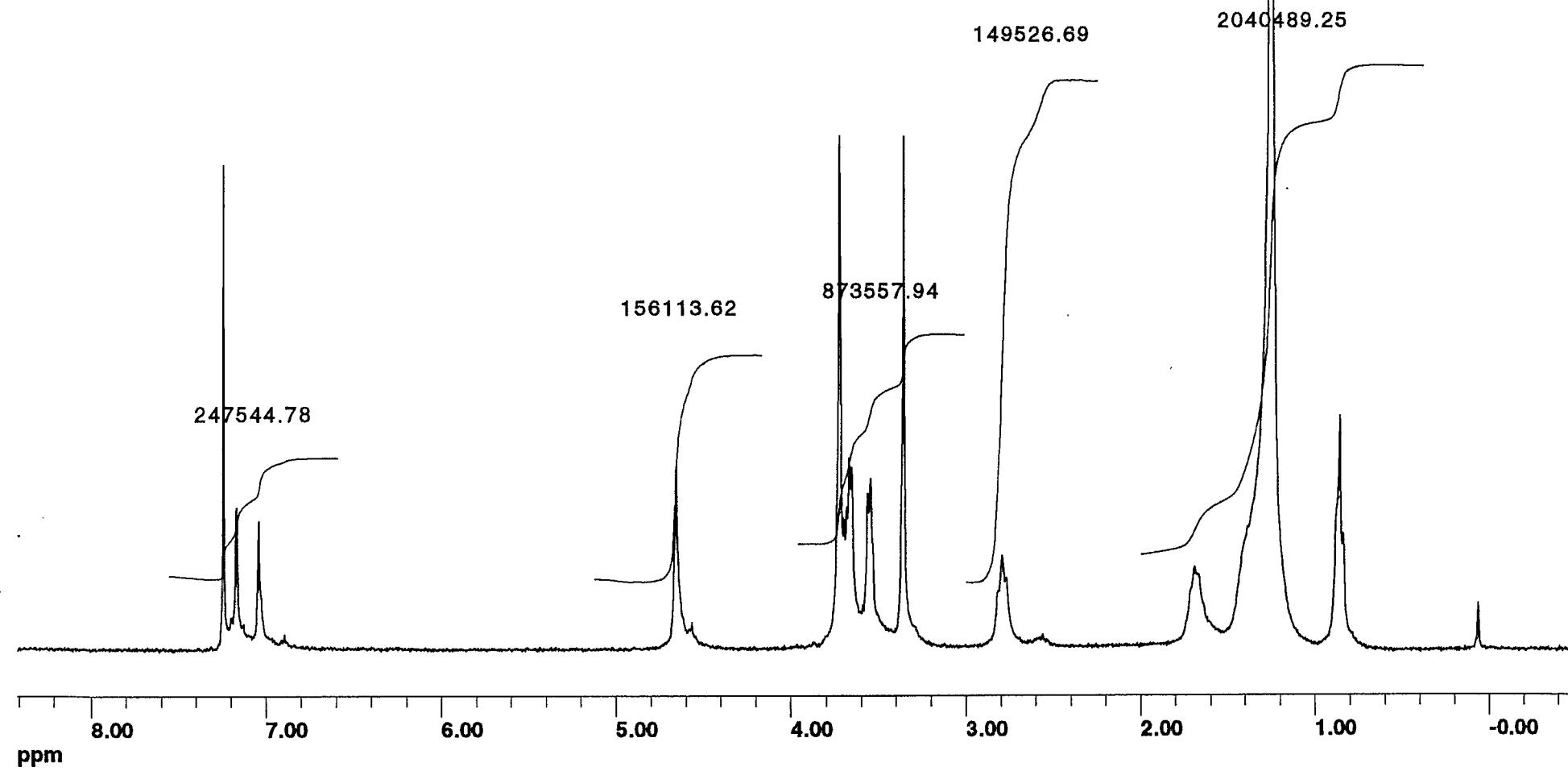
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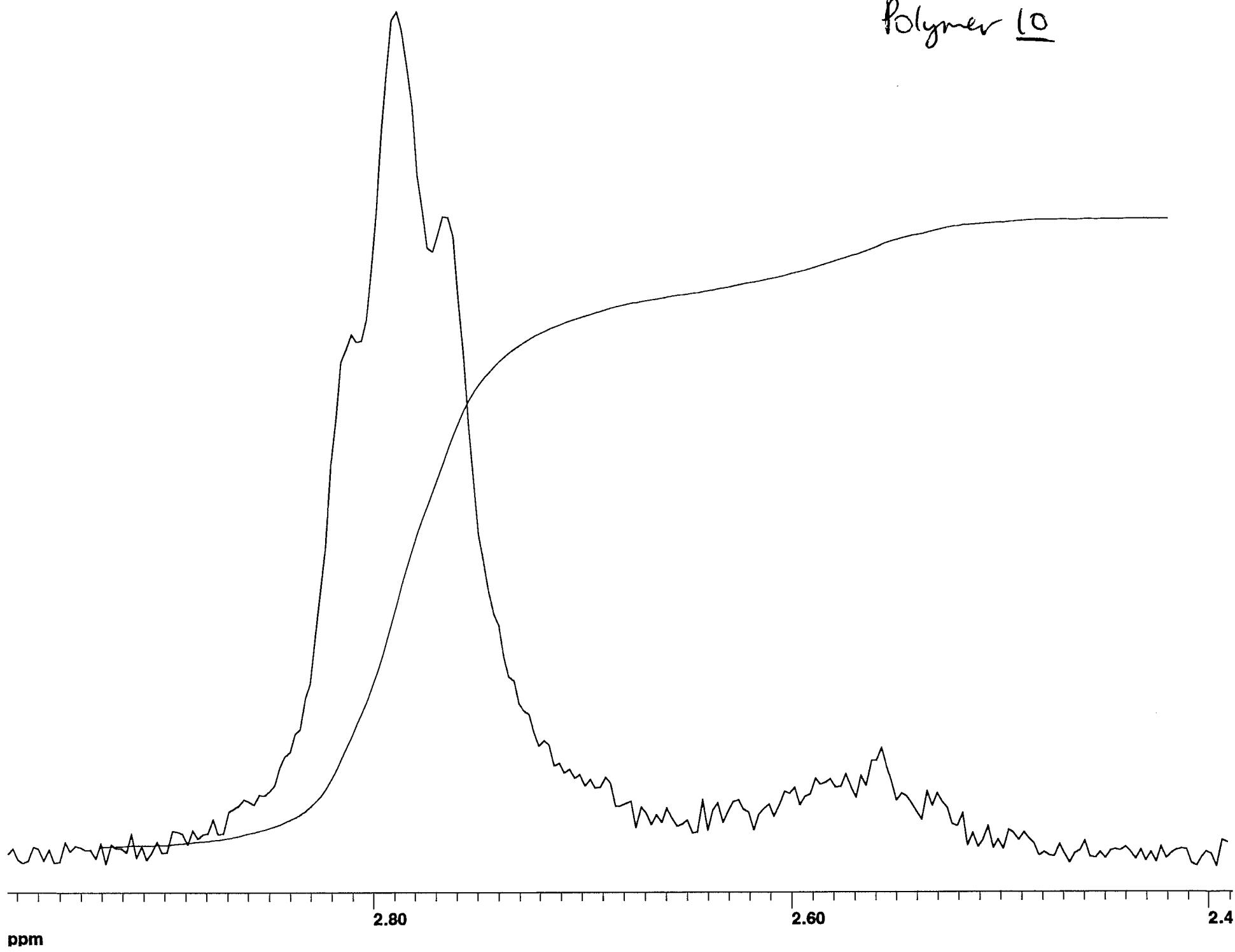
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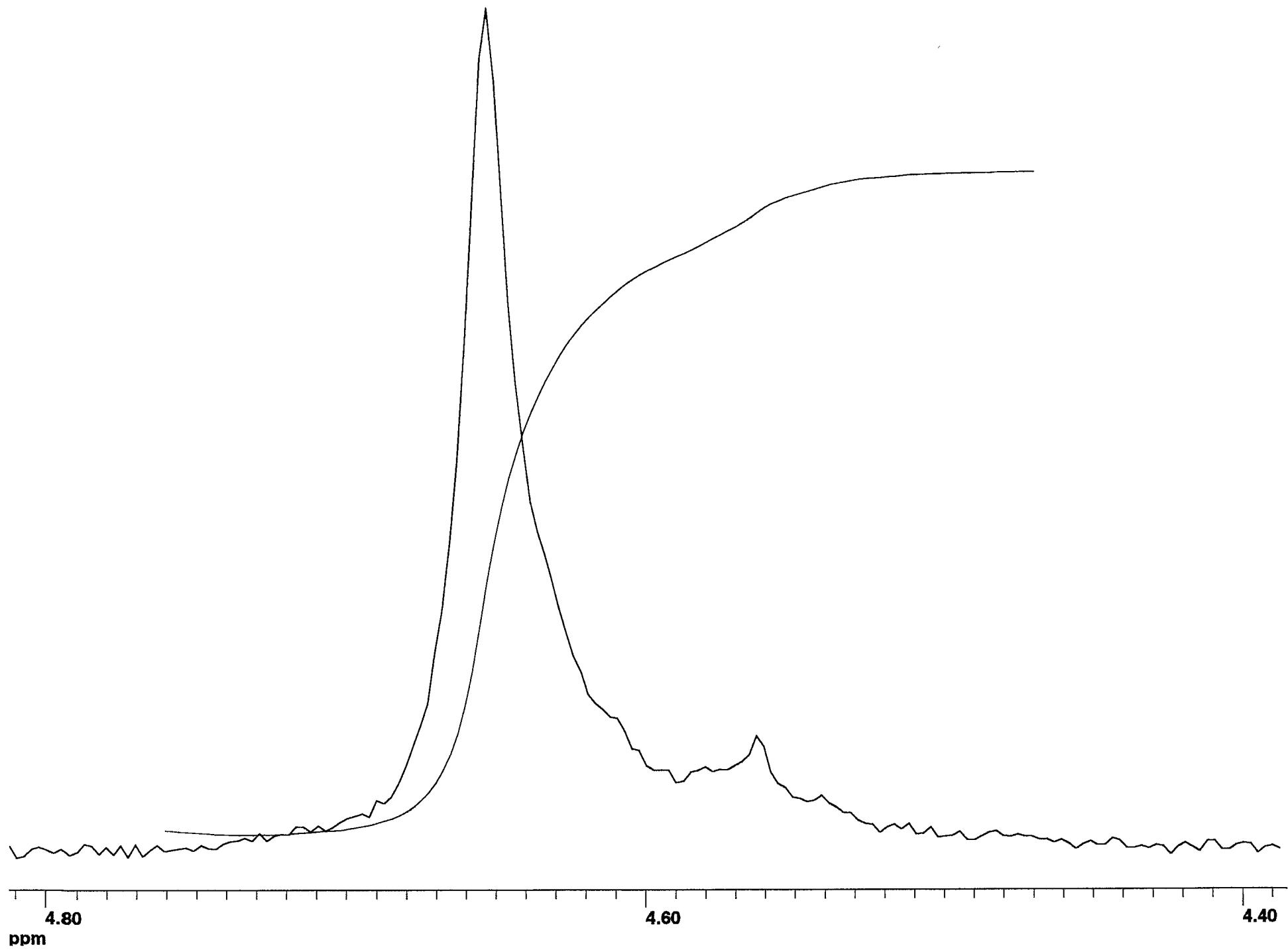


H NMR of MEEM-Dodecyl polymer





Polymer 10



Polymer 10

