

## Synthesis and Optical Recording Properties of Coupled Hemicyanine Salts for DVD-R

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A series of coupled-hemicyanine dyes with alkylene spacer were successfully synthesized by a reaction of coupled aldehydes with corresponding salts, respectively. These coupled dyes had more excellent thermal properties (high decomposition temperature, stiff decomposition behavior) and higher molar absorption properties than an uncoupled dye. The coupled dyes with perchlorate anions showed the strongest exothermic decomposition while those with hexafluorophosphate anions showed endothermic decomposition. As the coupling length ( $n=3, 4, 5, 6$ ) increased, thermal properties decreased and dyes with even spacer was more thermally stable than dyes with odd spacer. Among several coupled dyes, C4-NP-CIO<sub>4</sub> and C4-Cl-CIO<sub>4</sub> exhibited the best recording properties with the lowest jitter value of 7.5–9.5% in authoring disc.

**key words:** coupled hemicyanine dyes, optical disc, DVD-R, recording properties

### INTRODUCTION

Digital storage media with higher density have been demanded in growing information technology. Among a variety of storage media, especially, dye-based optical write once media, such as compact disc-recordable (CD-R), have a critical advantage because high capacity and low cost storage can be achieved compared to magnetic memory system, even though it is mainly for write-only read memory (WORM). As a newer optical recording system, Min et al have developed a metal polymer deformation (MPD) CD-R. [1] Since the standardization of the 3.95 GB digital versatile disc-recordable (DVD-R) has been completed and its commercialization was started in 1997, the 4.7 GB DVD-R have been also already established by these field industries, and compact disc (CD) system is replacing by DVD system at computers, games, movies, and electronic appliance [2].

The organic dyes for the recording layer of DVD-R require high molar absorption coefficients for sensitivity and readability at laser wavelength (635 or 658 nm) and fast thermogravimetric decomposition for desirable pit morphology. Also, they must have high thermal and photochemical stability, and good solubility in a specific solvent such as trifluoropropanol, which is a non-solvent for polycarbonate substrate. Some representative DVD-R dyes are azo/metal complex-type, cyanine-type, and styryl dyes [3–5]. Among these, we have been focused on the new styryl-type dyes because they can be easily synthesized and be used

in various potential applications [6,7].

For DVD-R dyes, it is very important to design the dyes having suitable UV-visible absorption spectra because they should be decomposed by an explosive heat derived from the DVD-R laser absorption. On the other hand, another requirement for DVD-R dyes is a good thermal stability. It means that dyes should be stable by a reading laser (650 nm) or sunlight, on the other hand, it should be decomposed very quickly at a certain of temperature and a certain of laser (635 or 658 nm). The anti-ion plays an important role to control exothermic/endothermic thermal decomposition of these DVD-R dyes. We have reported some styryl (hemicyanine) system for dye-based WORM, and investigated the effect of anti-ion on their spectral and thermal properties by means of ultraviolet-visible spectroscopy and thermal analysis measurements [8]. In this study, we designed a coupled hemicyanine-type dye system with different coupling spacer lengths for a search of more thermal stable and more adaptable DVD-R dyes. We investigated their thermal and optical properties, and recording properties using a commercial drive such as pioneer DVR S201 or DVR 103. We discuss herein the relationships between chemical structures (especially depending on spacer length) and recording properties for DVD-R.

### MATERIALS AND METHODS

#### *Measurements*

Absorption spectra on polycarbonate substrate and in trifluoropropanol solution were recorded on Jasco V-530 ultraviolet-visible spectrophotometer. <sup>1</sup>H-NMR spectra were taken with a 200 MHz Gemini, using CDCl<sub>3</sub> or DMSO-*d*<sub>6</sub> as a

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solvent. Thermogravimetric analysis (TGA) plots were taken on TA Instruments TGA 2950. The coupled hemicyanine dyes were dissolved in trifluoropropanol and coated on DVD-R polycarbonate substrate by a spin coating method and then silver reflective layer were sputtered on the dye layer. These discs were bonded with 0.6 mm dummy disc. Recording layer thickness was optimized by scanning of spin coating program. Fabricated discs were recorded by a commercial drive such as pioneer DVR S201 or DVR 103, and optical properties of the recorded disc were evaluated by DDU-1000 system.

#### Starting materials

4-(Diethylamino)salicylaldehyde as a starting material was purchased from Tokyo Kasei Co and used without further purification. Coupling agents such as dibromopropane, dibromobutane, dibromopentane, dibromohexane were purchased from Acros Co. and used without further purification. Indolium salts with anti-ion were synthesized by a general method as shown in Figure 1. 1,1,2,3-Tetramethyl-1H-benz[e]indolium iodide, of 5-chloro-2-methylene-1,3,3-trimethylindoline(1), and 70 wt% perchloric acid was purchased from Aldrich.

#### Synthesis of dialdehyde (C3)

In a 2 L three-necked flask equipped with a magnetic stirrer and a reflux condenser, 75.00 g (0.388 mol) of 4-(diethylamino)salicylaldehyde, 600 mL of tetrahydrofuran (THF), 53.60 g of potassium hydroxide, and several drops of Aliquat<sup>®</sup>336 were added and stirred at 100°C. And then, 1,3-dibromopropane (39.00 g / 0.194 mol) was dropped into the mixture. This mixture was reacted at reflux for 24 hours and then the cooled mixture was poured into cold water. The precipitate was filtered, dried, and purified by recrystallization from ethyl acetate to give 65.00 g (80% yield) of a desired product as yellow crystals (mp 143°C): <sup>1</sup>H-NMR (DMSO-*d*<sub>6</sub>); δ (ppm) 10.03 (2H, s), 7.49 (2H, d), 6.33 (2H, d), 6.17 (2H, s), 4.29 (4H, t), 3.44 (8H, m), 2.27 (2H, m), and 1.11 (12H, t).

The synthetic procedures (C4, C5) were used as the same method to C3.

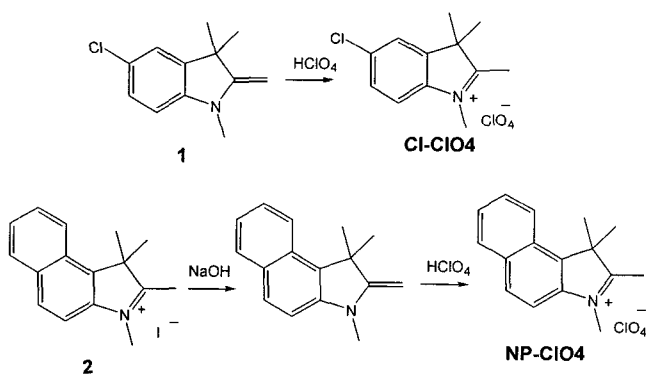


Figure 1. Synthesis of indolium salts.

C4: 93% yield, mp 140°C at first heating of DSC: <sup>1</sup>H-NMR (CDCl<sub>3</sub>); δ (ppm) 10.18 (2H, s), 7.72 (2H, d), 6.31 (2H, d), 6.04 (2H, s), 4.14 (4H, t), 3.43 (8H, m), 2.01 (4H, m), and 1.22 (12H, m).

C5: 95% yield, mp 123°C at first heating of DSC: <sup>1</sup>H-NMR (CDCl<sub>3</sub>); δ (ppm) 10.18 (2H, s), 7.72 (2H, d), 6.28 (2H, d), 6.03 (2H, s), 4.07 (4H, t), 3.43 (8H, m), 1.94 (4H, m), 1.73 (2H, m), and 1.22 (12H, t).

C6: 94% yield, mp 155°C at first heating of DSC: <sup>1</sup>H-NMR (CDCl<sub>3</sub>); δ (ppm) 10.19 (2H, s), 7.72 (2H, d), 6.28 (2H, d), 6.02 (2H, s), 4.06 (4H, t), 3.43 (8H, m), 1.86 (4H, m), 1.58 (4H, m), and 1.22 (12H, t).

#### Synthesis of indolium salt (Cl-C1O4)

Into 500 mL flask added 11.40 g (0.055 mol) of 5-chloro-2-methylene-1,3,3-trimethylindoline(1) and 300 mL of diethyl ether. The portion of 70 wt% perchloric acid solution (15.79 g, 0.11 mol) was slowly added dropwise into the solution and stirred for 5 h at room temperature. The precipitated white crystals was separated by filtration and washed with diethyl ether. The crystals were purified by recrystallization from ethanol.

#### Synthesis of indolium salt (NP-C1O4)

Into 500 mL flask added 1,1,2,3-tetramethyl-1H-benz[e]indolium iodide of 2 compound (19.30 g, 0.055 mol) and 300 mL of methanol. Into this solution, alkaline solution (40.00 g NaOH and 500 mL water) was added slowly and stirred for 2 h. This mixture was extracted with diethyl ether. Into the organic solution, added slowly 70 wt% perchloric acid solution (15.79 g, 0.11 mol). The precipitated white crystal was separated by filtration and washed with diethyl ether. The crystals were purified by recrystallization from ethanol.

#### Synthesis of dye (C3-Cl-C1O4)

Into 500 mL 1-neck flask added 0.93 g (3 mmol) of indolium salt (Cl-C1O4) and 300 mL of ethanol and then stirred at reflux for 3 days. Precipitated crystals were separated by filtration and washed with ethanol at reflux twice, and then separated by filtration. The final product was purified by recrystallization from acetonitrile at low temperature to give dark violet crystals. Yield 0.89 g (88%), <sup>1</sup>H-NMR(DMSO-*d*<sub>6</sub>): 1.14 (t, 12H), 1.64 (s, 12H, (CH<sub>3</sub>)<sub>2</sub>), 2.07 (m, 2H), 3.55 (q, 8H), 3.78 (s, 6H, N<sup>+</sup>-CH<sub>3</sub>), 4.45 (t, 4H), 6.27 (s, 2H), 6.60 (d, 2H), 7.12 (d, 2H), 7.57 (d, 2H), 7.59 (d, 2H), 7.79 (s, 2H), 8.04 (d, 2H), 8.39 (d, 2H).

The other dyes were synthesized in the same manner as described above.

C4-Cl-C1O4; Yield 0.37 g (36%), <sup>1</sup>H-NMR(DMSO-*d*<sub>6</sub>): 1.16 (t, 12H), 1.63 (s, 12H, (CH<sub>3</sub>)<sub>2</sub>), 2.01 (m, 4H), 3.57 (q, 8H), 3.78 (s, 6H, N<sup>+</sup>-CH<sub>3</sub>), 4.27 (t, 4H), 6.26 (s, 2H), 6.58 (d, 2H), 7.11 (d, 2H), 7.58-7.60 (m, 4H), 7.74 (s, 2H), 8.02 (d, 2H), 8.39 (d, 2H).

C5-Cl-C1O4; Yield 0.87 g (84%), <sup>1</sup>H-NMR(DMSO-*d*<sub>6</sub>): 1.17 (t, 12H), 1.65 (s, 12H, (CH<sub>3</sub>)<sub>2</sub>), 1.86 (m, 2H), 2.01 (m, 4H), 3.59 (q, 8H), 3.80 (s, 6H, N<sup>+</sup>-CH<sub>3</sub>), 4.29 (t, 4H), 6.28 (s, 2H), 6.60 (d,

2H), 7.17 (d, 2H), 7.60-7.63 (m, 4H), 7.76 (s, 2H), 8.03 (d, 2H), 8.41 (d, 2H).

C6-Cl-CIO<sub>4</sub>; Yield 0.96 g (91%), <sup>1</sup>H-NMR(DMSO-*d*<sub>6</sub>): 1.13 (t, 12H), 1.63 (s, 12H, (CH<sub>3</sub>)<sub>2</sub>), 1.70 (m, 4H), 1.92 (m, 4H), 3.53 (q, 8H), 3.78 (s, 6H, N<sup>+</sup>-CH<sub>3</sub>), 4.22 (t, 4H), 6.20 (s, 2H), 6.53 (d, 2H), 7.10 (d, 2H), 7.57 (d, 2H), 7.63 (s, 2H), 7.80 (s, 2H), 8.00 (d, 2H), 8.41 (d, 2H).

C3-NP-CIO<sub>4</sub>; Yield 0.79 g (76%), <sup>1</sup>H-NMR(DMSO-*d*<sub>6</sub>): 1.15 (t, 12H), 1.85 (s, 12H, (CH<sub>3</sub>)<sub>2</sub>), 2.61 (m, 2H), 3.59 (q, 8H), 3.98 (s, 6H, N<sup>+</sup>-CH<sub>3</sub>), 4.58 (t, 4H), 6.43 (s, 2H), 6.66 (d, 2H), 7.26 (d, 2H), 7.60 (t, 2H), 7.70 (t, 2H), 7.90 (d, 2H), 8.10-8.21 (m, 8H), 8.60 (d, 2H).

C4-NP-CIO<sub>4</sub>; Yield 0.93 g (88%), <sup>1</sup>H-NMR(DMSO-*d*<sub>6</sub>): 1.21 (t, 12H), 1.89 (s, 12H, (CH<sub>3</sub>)<sub>2</sub>), 2.26 (m, 4H), 3.63 (q, 8H), 3.96 (s, 6H, N<sup>+</sup>-CH<sub>3</sub>), 4.44 (t, 4H), 6.40 (s, 2H), 6.65 (d, 2H), 7.22 (d, 2H), 7.57-7.60 (m, 4H), 7.88 (d, 2H), 8.11-8.19(m, 8H), 8.60 (d, 2H).

C5-NP-CIO<sub>4</sub>; Yield 0.29 g (27%), <sup>1</sup>H-NMR(DMSO-*d*<sub>6</sub>): 1.12 (t, 12H), 1.88 (s, 12H, (CH<sub>3</sub>)<sub>2</sub>), 1.95 (m, 2H), 2.10 (m, 4H), 3.52 (q, 8H), 3.96 (s, 6H, N<sup>+</sup>-CH<sub>3</sub>), 4.36 (t, 4H), 6.32 (s, 2H), 6.51 (d, 2H), 7.19 (d, 2H), 7.58-7.62 (m, 4H), 7.91 (d, 2H), 8.06-8.20 (m, 8H), 8.52 (d, 2H).

## RESULTS AND DISCUSSION

As shown in Figure 1, indolium salts were easily prepared by a reaction of trimethylindoline derivative with perchloric acid. The coupled dialdehyde C<sub>n</sub> (n=3, 4, 5, 6) could be synthesized by a condensation of 4-(diethylamino)salicylaldehyde with dibromoalkyls. Tetrahydrofuran as solvent and potassium hydroxide as base gave the best coupling conversion. Also, addition of a phase transfer agent such as Aliquat®336 helped the reaction. Figure 2 displayed the synthesis of dyes used in this study. The coupled hemicyanine dyes (C<sub>n</sub>-Cl-CIO<sub>4</sub> or C<sub>n</sub>-NP-CIO<sub>4</sub>) were synthesized by a reaction of C<sub>n</sub> and corresponding indolium salts (Cl-CIO<sub>4</sub>, NP-CIO<sub>4</sub> in Figure 1), respectively. Since the dialdehyde C<sub>n</sub> had two reaction sites (dialdehydes) with salts, there was always one-site reaction adduct in reaction medium together with two-site reaction product. Therefore, the reaction must be continued until one-site pink-fluorescent spot on thin-layer chromatography (TLC) was disappeared, while checking through TLC. The final product was precipitated in a crystal form in reaction medium. Because one-site reaction adduct had better solubility in ethanol than two-site product, it could

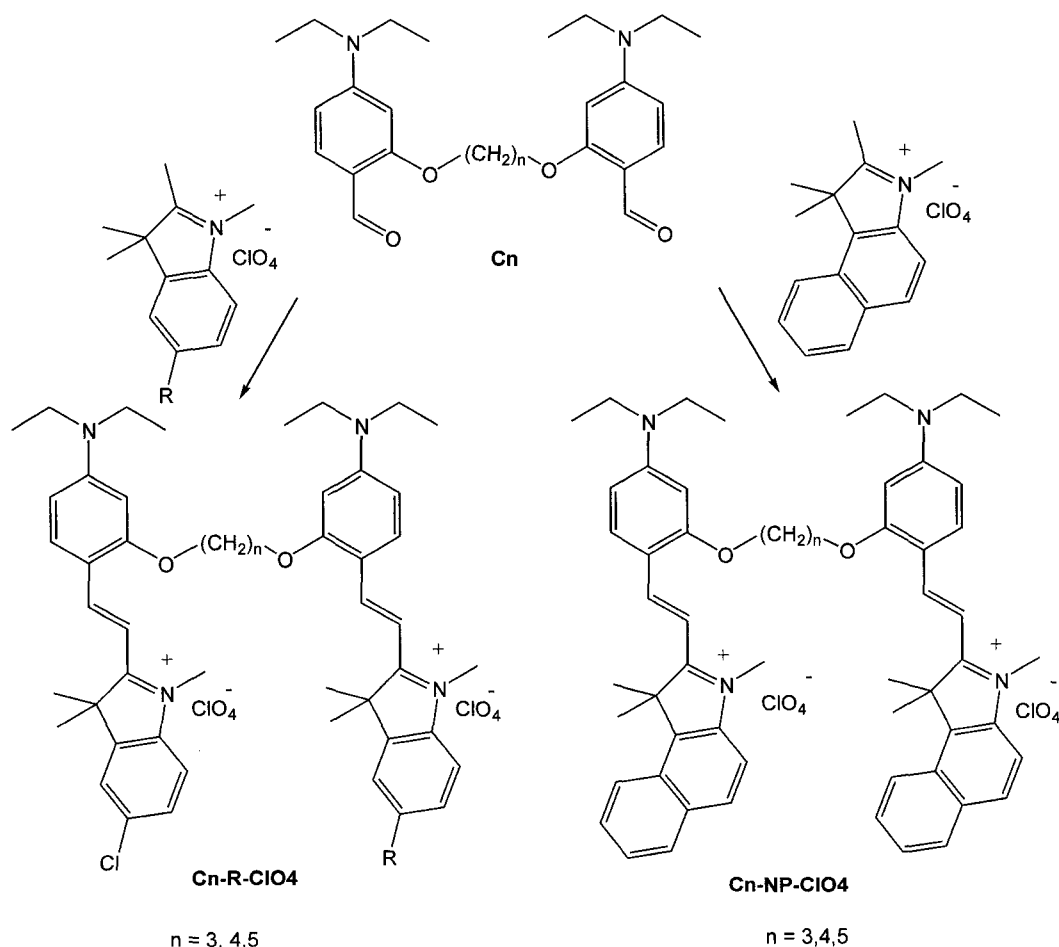


Figure 2. Synthesis of coupled hemicyanines.

be removed by extraction with hot ethanol one or two times. The coupled dye product was hardly soluble even in hot ethanol, therefore, it was easy to separate by filtration. All dye products were highly soluble in both trifluoropropanol and

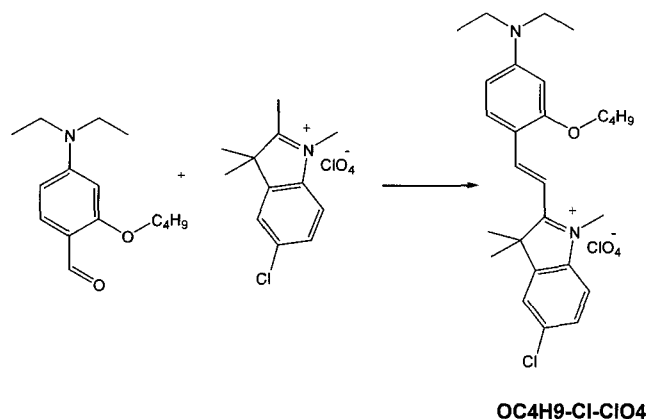


Figure 3. Synthesis of an uncoupled hemicyanine.

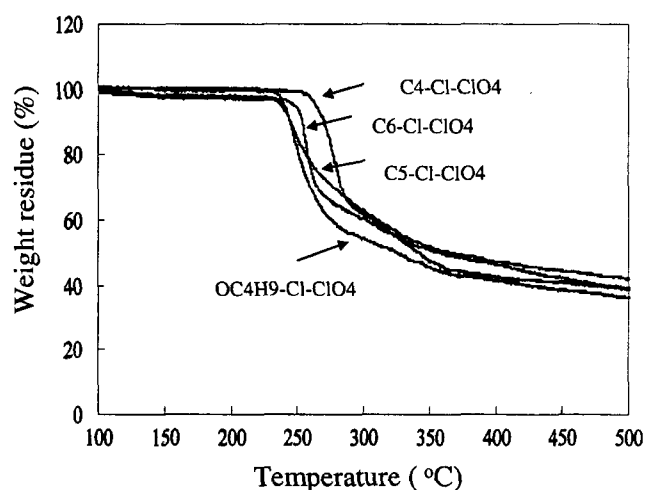


Figure 4. TG curves of coupled hemicyanines (Cn-Cl-C1O4) and an uncoupled hemicyanine.

acetonitrile. The structure of all products were well characterized by  $^1\text{H-NMR}$  spectroscopy.

Figure 3 shows the representative thermogravimetric curves of three coupled hemicyanines (Cn-Cl-C1O4) and an uncoupled hemicyanine (OC4H9-Cl-C1O4). Coupled cyanines had an about 30°C higher initial decomposition temperature than an uncoupled one, implying that this coupling method was very effective to enhance the thermal stability of DVD-R dyes material. The thermal stability was in the order of C3=C4>C6>C5, meaning that dyes with shorter spacer and even alkylene spacer had more stable than those with longer spacer and odd alkylene spacer. Also, the thermal decomposition behavior of C4-Cl-C1O4 dye was stiffer than C5-Cl-C1O4, which must be one of desirable properties for DVD-R dyes to make distinct pit morphology. As shown in differential temperature analysis (DTA) of Figure 4, all coupled dyes did not show any melting points while an uncoupled dye (OC4H9-Cl-C1O4) had a melting point (endothermic) at 210°C. The Cn-Cl-PF6 and Cn-NP-PF6 showed all endothermic decomposition, among these, C4-Cl-C1O4 exhibited the highest exothermic decomposition around 280°C. From these results, we could expect that C4-Cl-C1O4 would show the best recording properties in our dyes.

The thermal and optical properties of dyes were summarized in Table 1. The maximum absorption wavelengths ( $\lambda_{\text{max}}$ ) were determined by UV-visible spectroscopy on polycarbonate substrate and in tetrafluoropropanol solution, respectively. The  $\lambda_{\text{max}}$  of coupled dyes on substrate had in the range of 590–600 nm and the molar absorption coefficients were as high as adaptable for DVD-R dyes. The Cn-Cl-C1O4 series with chlorobenzene unit had higher molar absorption coefficients (260,000–280,000) than Cn-NP-C1O4 series with naphthalene unit. The high molar absorption coefficients were attributed to two dye molecules in a molecule.

A coupled dye was dissolved in trifluoropropanol and coated on DVD-R polycarbonate substrate by a spin coating method and then silver reflective layer were sputtered on the dye layer. Discs with Cn-NP-C1O4 were recorded by

Table 1. General properties of coupled dyes

Dye	$\lambda_{\text{max}}$ (nm)		Molar absorption coefficient	$T_m^c$ (°C)	$T_d^d$ (°C)
	on polycarbonate <sup>a)</sup>	in TFP <sup>b)</sup>			
OC4H9-Cl-C1O4	592	556	130,000	210	245
C3-Cl-C1O4	594	556	261,000	-	272
C4-Cl-C1O4	593	560	76,000	-	265
C5-Cl-C1O4	593	554	281,000	-	244
C3-NP-C1O4	596	570	195,000	0	293
C4-NP-C1O4	597	566	187,000	-	284
C5-NP-C1O4	597	569	190,000	-	266

<sup>a)</sup> Measured on polycarbonate substrate by a reflection mode of UV-visible spectroscopy.

<sup>b)</sup> Measured in trifluoropropanol solution by a transmission mode of UV-visible spectroscopy.

<sup>c)</sup> Melting points were measured by DTA at a heating rate of 10 °C/min under nitrogen.

<sup>d)</sup> Initial decomposition temperatures were measured by TG at a heating rate of 10 °C/min under nitrogen.

Table 2. Recording properties of Cn-NP-CIO4

	General (658 nm recording)				Authoring (635 nm recording)			
	X <sup>a)</sup>	R <sub>top</sub> <sup>b)</sup> (%)	Jitter (%)	I <sub>14</sub> /I <sub>14H</sub> <sup>c)</sup>	X <sup>a)</sup>	R <sub>top</sub> <sup>b)</sup> (%)	Jitter (%)	I <sub>14</sub> /I <sub>14H</sub> <sup>c)</sup>
C3-NP-CIO4	3	47.6	10.6	0.59	3	50.2	10.9	0.64
	4	49.6	11.1	0.63	4	48.4	14.3	0.66
	5	51.3	11.2	0.58	5	51.9	11.2	0.58
C4-NP-CIO4	3	50.7	9.6	0.55	3	51.3	9.5	0.56
	4	51.3	10.1	0.55	4	-	-	-
	5	53.7	12.1	0.53	5	53.1	11.9	0.58
C5-NP-CIO4	3	50.7	11.0	0.56	3	-	-	-
	4	-	-	-	4	51.9	12.5	0.60
	5	63.1	12.3	0.55	5	53.1	12.5	0.59

<sup>a)</sup>Spin coating program

<sup>b)</sup>Top reflectivity

<sup>c)</sup>Maximum modulated intensity

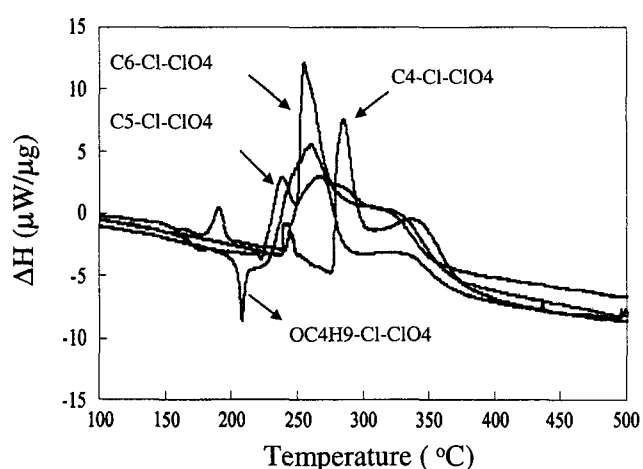


Figure 5. DTA curves of coupled hemicyanines (Cn-Cl-CIO4) and an uncoupled hemicyanine.

authoring (635 nm recording) and general drive (658 nm recording) at DDU-1000 system. Table 2 shows the recorded optical properties at DDU-1000 system. The properties depend on the dye layer thickness controlled by spin coating program, X=3, 4, and 5. The C3-NP-CIO4 disc showed the highest I<sub>14</sub>/I<sub>14H</sub> signal amplitude. These results seem to be derived from the highest molar absorption coefficient and decomposition temperature which induce the highest optical path difference and the largest deformation of substrate. On the other hand, C4-NP-CIO4 showed the lowest jitter value of 9.5%. This phenomenon was explained by sharp decomposition of dye which induce sharp pit edge. The C4-NP-CIO4 disc had the smaller I<sub>14</sub>/I<sub>14H</sub> due to lower molar absorption coefficient and decomposition temperature compared to C3-NP-CIO4. Based on Table 2, however, the C4-NP-CIO4 would be the best candidate in this study taking account to low jitter value together with high reflectance (R<sub>top</sub>).

The coupled dyes, Cn-Cl-CIO4 series exhibited somewhat

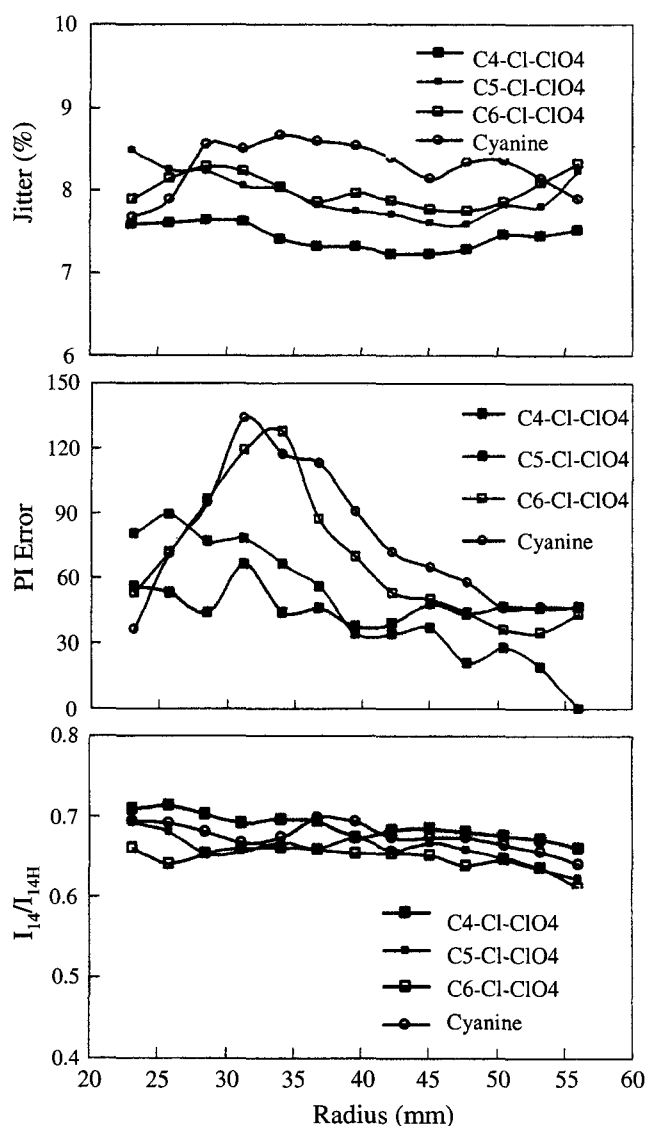


Figure 6. Authoring recording properties of Cn-Cl-CIO4 depending on polycarbonate substrate radius.

better recording properties at authoring rather than general drive recording. Correspondingly, C<sub>n</sub>-Cl-CIO<sub>4</sub> dyes (n=3, 4, 5) were also applied to DVD-R authoring and evaluated by a commercial drive such as pioneer DVR S201 or DVR 103. The optical recording properties of the disc were compared with a disc made by a commercial cyanine dye. Figure 6 shows the recording properties (jitter, PI error, and  $I_{14}/I_{14H}$ ) depending on polycarbonate substrate radius. This result reflects how uniform their recording properties are on the disc substrate. Among coupled dyes, C4-Cl-CIO<sub>4</sub> disc has the best properties because it had the lowest jitter value (7.5 %), the lowest PI error rate, and the highest  $I_{14}/I_{14H}$ . The results would be responsible for the highest decomposition temperature with sharp decomposition pattern as shown in Figure 5. Conclusively, C4-Cl-CIO<sub>4</sub> disc had the better optical properties than a commercial cyanine disc.

### CONCLUSIONS

For an objective to enhance thermal stability of DVD-R dyes, we synthesized several coupled hemicyanines containing two DVD-R dyes in a molecule. We could observe the advanced thermal decomposition behavior of coupled dyes by means of thermogravimetric and differential temperature analysis. Dyes with ClO<sub>4</sub><sup>-</sup> anit-ion exhibited an exothermic decomposition while those with PF<sub>6</sub><sup>-</sup> anti-ions exhibited an endothermic decomposition. The thermal properties were also related with the coupling spacer length (n=3, 4, 5, 6). As the coupling length increased, thermal properties decreased and dyes with even alkylene spacer was more thermally stable than dyes with odd alkylene spacer. Among several coupled dyes in this study, C4-NP-CIO<sub>4</sub> and

C4-Cl-CIO<sub>4</sub> exhibited the lowest jitter value of 7.5~9.5% in authoring disc.

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