CONCLUSIONS

The significant differences in the racemization rates of amino acids and the corresponding amides make it possible to carry out selective racemization of amides in the presence of optically active amino acids.

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REACTION OF AMINALS OF CONJUGATED ω-DIMETHYLAMINO ALDEHYDES WITH INDANDIONE

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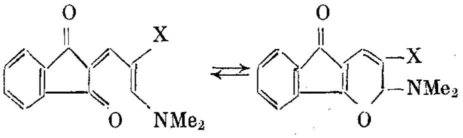
UDC 542.91:547.447.5:547.665

In a continuation of our research on δ-aminodienone $\rightleftarrows$ 2-amino-2H-pyran valence isomerization, in the present paper we have studied the condensation of aminals $Me_2NCH{=}C(X)CH(NMe_2)_2$ [I, X = H (a), Me (b), Cl (c)] and $Me_2N(CH{=}CH)_nCH(NMe_2)_2$ [I, n = 2 (d), 3 (e), 4 (f)] with indandione in order to obtain new δ-dimethylaminodienones IID and investigate their valence isomerization to the corresponding 2-amino-2H-pyrans IIP.



(II) D (II) P

For aminodienones IID we expected a shift of the absorption maximum in the electronic spectra to the longer-wave region as compared with aminodienones that do not contain an aromatic ring [1-3], which is important for practical purposes in connection with the fact that compounds of this type have solvato-, thermo-, and photochromic properties.

In contrast to the numerous examples of the condensation of aminals with acyclic β-dicarbonyl compounds and ketones [2, 3], which generally leads to a single product, the reaction of aminals with indandione proceeds ambiguously. Thus, Ia, b react with indandione in benzene at 20°C to give mixtures of products, from which, by preparative thin-layer chromatography (TLC), we isolated crystalline orange compounds, which were identified as δ-aminodienones IIa and IIb from PMR, mass, and UV spectral data and the results of elementary analysis. Pure IIa is obtained in 23% yield by condensation of indandione with 1,1,3-trimethoxy-3-dimethylaminopropane in absolute ether. Compound IIc was obtained as the only product in 42% yield by condensation of indandione with Ic in benzene.

A comparison of the absorption curves of IIa,b,c in various solvents (EtOH, $CHCl_3$, hexane) showed that these compounds exist only in open dienone form D and do not undergo cyclization to 2-amino-2H-pyrans.

N. D. Zelinskii Institute of Organic Chemistry, Academy of Sciences of the USSR, Moscow. Translated from Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No. 7, pp. 1596-1602, July, 1986. Original article submitted January 2, 1985.

TABLE 1. ^{13}C NMR Spectra of IIIa, IVa, IVb, and XIa in d_6-DMSO at ∿25°C (δ, ppm)[a]

Compound	Cation					Anion				
	α′	β′	γ′	NMe_2	C=O	α	β	γ	Me	Ph
(IIIa)[b]	162,8 d	90 d	162,8 d	37,8 q 45,6 q	190,5	114,0	148,4	114		141,1, 139,9 (C′), 133,1 &120,5 (*o*-, *m*-)
(IVa)					189,3	113,0	147,3	113		140,2, 139,6 (C′), 132,0 &119,3 (*o*-, *m*-)
(IVb)					189,4	114,2	152,0	124,2	19,4	140,2 (C′), 132,4& 120,1 (*o*-, *m*-)
(XIa)					189,2	113,5	132,6			140,6 (C′),[c] 132,4 & 120,4 (*o*-, *m*-)

Data from Monoresonance Spectra for IVa, IVb, and XIa

Carbon atom (compound)	Spectral form	J[d], Hz
C=O (IVa), (IVб)	d	$^3J=6$ (H_β) or H-*o*
C=O (XIa)	m[e]	
α-(IVa)	d	$^3J=3$ (H_γ)
α-(IVb)	s	
α-(XIa)	d	$^2J=2$ (H_β)
β-(IVa)	dd	$^1J=152$, $^2J=8$ (H_γ)
β-(IVb)	dm[e]	$^1J=150$
β-(XIa)	d	$^1J=148$
γ-(IVa)	dm[e]	$^1J=170$
C′ (IVa), (IVb), (XI)	m	
γ-(IVb)	m	
o-, *m*-(IVa), (IVb), (XI)	dm[e]	$^1J=163-165$
Me (IVb)	qm[e]	$^1J=128$

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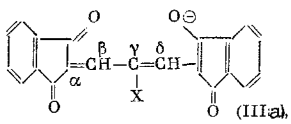
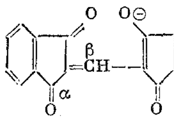
[a] Designation of the C atoms in the



and

(IIIa), (IVa), (IVb)



(XIa)

anions and in the $Me_2N-\overset{\alpha'}{C}H=\overset{\beta'}{C}H-\overset{\gamma'}{C}H=NMe_2$. cation.

[b] In the assignment, data from the spectrum obtained under "off-resonance" conditions were taken into account.

[c] Significantly broad signals in the double-resonance spectrum.

[d] The H atoms that couple with C atoms are indicated in parentheses.

[e] The precise forms of the multiplets and the corresponding J values were not determined because of the low signal/noise ratio.

In addition to aminodienones IIa and IIb, from the products of the reaction of indandione with Ia, b we isolated by twofold crystallization, individual, crystalline, bright-green trimethylidyneoxanine salts IIIa and IIIb, the structures of which were confirmed by the ^{13}C and ^{1}H NMR spectra (Tables 1 and 2), the electronic spectra, the results of elementary analysis, determination of the molecular masses (by ebullioscopy), and some chemical transformations.

TABLE 2. PMR Spectra (in d_6-DMSO)[a, b]

Compound	Cation						Anion						
	α'	β'	γ'	Me	NMe_2	$J_{\alpha,\beta}$, Hz	β	γ	δ	Me	Ph	J, Hz	
												β, γ	γ, δ
(IIIa)[c]	7,79	5,49	7,79		3,17 & 3,37	12	7,6	8,61	7,6		7,85	13[d]	15[d]
(III[b])	7,54		7,54	2,26	3,44		7,62		7,62	2,9	7,84		
(IV[b])							7,72		7,72	2,98	7,84		
(VIb)							7,65			2,85	7,85		
(IVa)							7,61		7,61		7,81	13[d]	15[d]
(VIa)							7,36	8,61	7,36		7,61	13,7	13,7
(XI)	7,5–7,85	5,33	7,5–7,85		3,02 & 3,22	12	7,5–7,85	8,36			7,5–7,85		
(XIa)							7,74				7,81		
(X)[e]							8,48				7,26–7,66		

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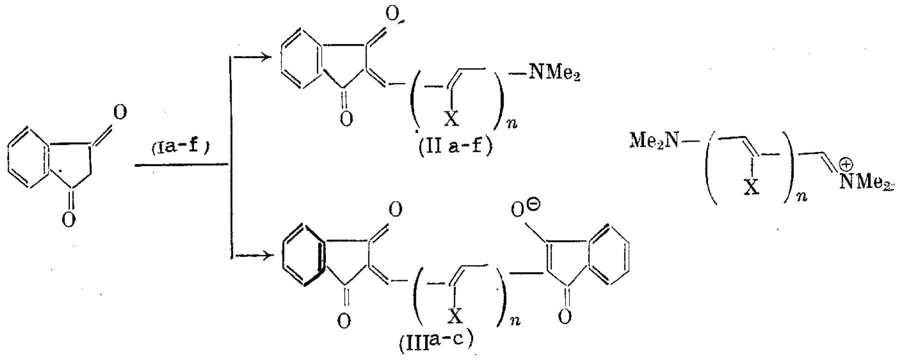
[a] The δ values in parts per million relative to hexamethyldisiloxane (HMDS) as the external standard.

[b] The designation of the atoms is indicated in Table 1.

[c] The assignment of the signals was made with allowance for double-resonance data with respect to the $H_{\beta'}$, Ph, and H_γ groups.

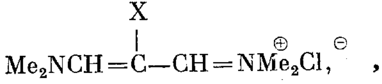
[d] The assignment of the J values is arbitrary.

[e] In C_6D_5N with respect to HMDS as the internal standard.

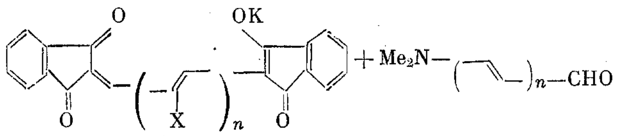


Under the influence of a 10% methanol solution of KOH salts IIIa and IIIb are converted quantitatively to slightly soluble oxanine K salts IVa and IVb and aldehydes Va and Vb, which remained in the mother liquors. This transformation is illustrated graphically by the electronic spectra: bands at 248 and 559 nm (IVa) and at 248 and 561 nm (IVb) are observed for the K salts; the spectra of the mother liquors contain bands at 285 and 290 nm, which correspond to aldehydes Va and Vb.

Under the influence of a 10% alcohol solution of HCl salts IIIa and IIIb form slightly soluble oxanines VIa* and VIb, which in EtOH have the same λ_{max} values as K salts IVa and IVb; chlorides



$Me_2NCH{=}\overset{X}{\overset{|}{C}}{-}CH{=}\overset{\oplus}{N}Me_2\overset{\ominus}{Cl}$, which have λ_{max} 312 and 324 nm, are found in the mother liquors.



(IVa,b,d) (Va, b)

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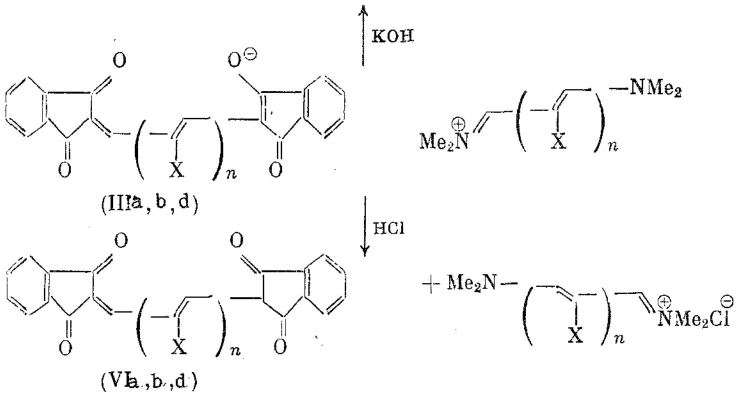
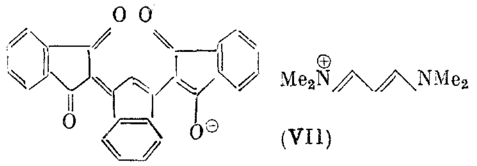
*Oxanine VIa and its trimethylammonium salt were obtained in [4] and [5].

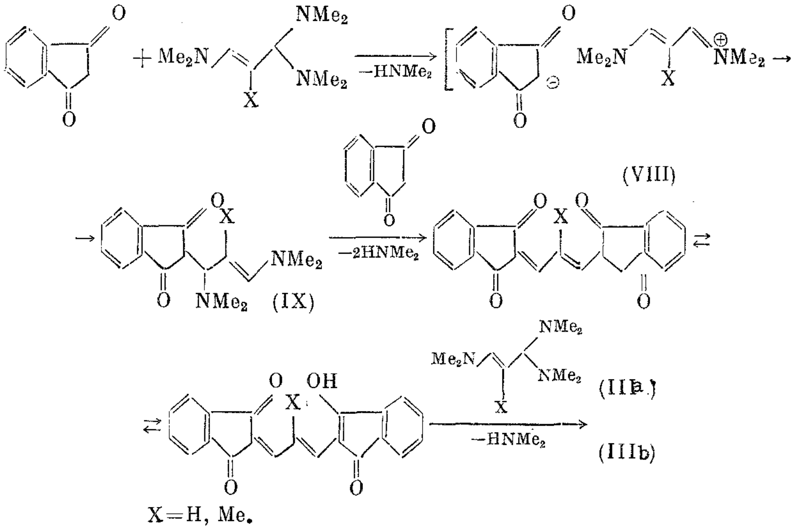


X = H, n = 1(IIIa) — (VIa); X = Me, n = 1 (IIIb) — (VIb); X = H, n = 2 (IVd), (IIId), (VId).

In addition to oxanine salt IIIa, a very small amount (<5%) of salt VII, the anion of which is the product of crotonic condensation of three indandione molecules, is formed:

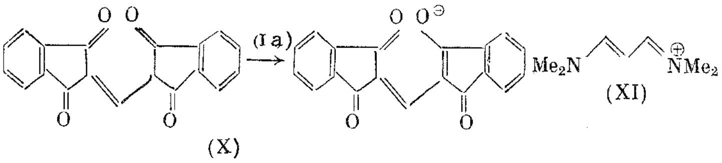

(VII)

The data obtained in [7] make it possible to present the following scheme for the synthesis of IIIa, b:



X=H, Me.

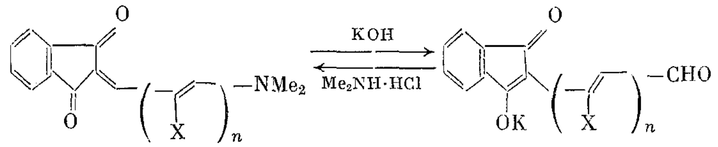
The possibility of the formation of salt VIII and diamine IX as intermediates is confirmed by the isolation of such products in the reactions of animals with compounds that contain an active CH_2 group. We have also previously observed replacement of the NMe_2 group by the anion of a CH acid in the reaction of Ia with $CH_2(CN)_2$, in which ketene imine $NH{=}C{=}C(CN)CH{=}CHCH{=}C(CN)_2$ was isolated. Oxanine IV readily undergoes enolization, which is confirmed by the identical absorption spectra of IV and VI in EtOH, and, finally, we have previously observed that animals are protonated extremely readily even upon dissolving in alcohols. This fact explains the conversion of VI to III. In fact, salt XI was isolated by the action of an animal on methylidyneoxanine X:



(X)
(XI)

We subsequently studied the condensation of indandione with Id-f [8]. We observed that a mixture of pentamethylidyneoxanine salt IIId and triene diketone IId in ratios* of 1:2 (when the reaction is carried out in benzene) and 4:1 (without a solvent) is formed from Id in ∿70% yield. Diketone IId is readily separated by crystallization; from the mother liquor, which contains primarily IIId, we isolated either IVd by the action of an alcohol solution of KOH or VId by the action of an alcohol solution of HCl.

Under the influence of an alcohol solution of KOH diketone IId is converted quantitatively to aldehyde IIIa, from which IId is again formed by treatment with $Me_2NH \cdot HCl$:



(IId), (IIb) (XIIa), (XII b)

$X = H$, $n = 2$ (IId), (XIIa); $X = Me$, $n = 1$ (IIb), (XIIb).

Similar transformations were observed for IIg (n = 0) [9], as well as IIb, from which XIIb was obtained.

Only polyene ω-dimethylamino diketones IIe and IIf are formed in 60% and 20% yields from aminals Ie, f. The structures of IId, e, f, as well as IVd, VId, XIIa, and XIIb, were confirmed by data from the electronic, PMR, and mass spectra and the results of elementary analysis.

It should be noted that, in contrast to known merocyanines [10-12], in conjugated ω-dimethylamino diketones IIg (n = 0) and IIa, d, e, f, as the length of the polymethine chain increases, the shift to the red region of each succeeding member does not decrease and amounts to ∿90-115 nm in the case of lengthening by one C=C bond. A similar principle was previously observed [12] in series of dinitriles $Me_2N(CH{=}CH)_nCH{=}C(CN)_2$, where n = 1-3.

EXPERIMENTAL

The UV spectra were recorded with a Specord UV-VIS spectrophotometer. The PMR spectra were obtained with Bruker WM-250 and Bruker BS-467 spectrometers (250 and 100 MHz). The ^{13}C NMR spectra were recorded with Bruker WM-250 and Bruker WP-60 spectrometers (62.89 and 15.08 MHz) with complete suppression of spin–spin coupling with the protons; the "off-resonance" spectrum was obtained for IIIa, and monoresonance spectra were obtained for IVa, IVb, and XIa. Solutions of the compounds in d_6-DMSO (∿0.4 M) were investigated; the internal standard was d_6-DMSO, the chemical shift of which was taken was 39.5 ppm.

Condensation of Indandione with Aminals Ia-c. A) A mixture of 0.86 g of indandione and 1.0 g of aminal Ia in 10 ml of dry benzene was maintained at 20-22°C for 4 h, after which the solvent was evaporated in vacuo, and the residual dark viscous oil [λ_{max} 248, 312, 440, and 559 nm (EtOH)] was washed with absolute ether and dissolved in acetone. The solution was cooled, and the resulting precipitate was recrystallized from acetone to give 0.43 g (32%) of IIIa in the form of dark-green crystals with mp 203-205°C. Found: C 74.22; H 5.91; N 6.37%. $C_{28}H_{26}N_2O_4$. Calculated: C 74.00; H 5.73; N 6.16%. Electronic spectrum [λ_{max}, nm (ε)] in EtOH: 248 (62,000), 312 (72,000), and 559 (134,000); In $CHCl_3$: 248 (40,000), 312 (40,900), and 575 (136,000).

Workup of the mother liquor gave 0.4 g of VII with mp 200-201°C. Found: C 77.08; H 5.48; N 5.23%. $C_{34}H_{28}N_2O_4$. Calculated: C 77.26; H 5.31; N 5.30%. Electronic spectrum [λ_{max}, nm (ε)] in EtOH: 268 (50,980), 312 (53,500), 422 (21,300), 670 (20,700), 720 (38.700), and 785 (35,000). PMR spectrum (d_6-DMSO, δ,ppm) of the cation: 3.32 and 3.11 (12H, NMe_2), 7.68 (2H, H_α and H_γ), and 5.38 (1H, H_β); of the anion: 7.86, 7.44, and 9.22 (12H, Ph) and 8.94 (1H, CH).

A 1-ml sample of a 10% ethanol solution of HCl was added to a solution of 0.03 g of VII in 1 ml of EtOH, and the mixture was worked up to give 0.015 g of VIIa with mp 336°C (see [6]). The evaporated mother liquors, after separation of IIIa and VII, were subjected

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*The ratios of IId and IId were estimated from the electronic spectra.

to preparative thin-layer chromatography (TLC) on SiO_2 in an acetone–hexane system (1:1) by elution of the bright-yellow zone with $CHCl_3$ to give 0.4 g (30%) of IIa in the form of a yellow precipitate with mp 228-230°C (from MeOH). Found: C 74.22; H 5.91; N 5.85%. $C_{14}H_{13}NO_2$. Calculated: C 74.00, H 5.73; N 6.16%. UV spectrum [λ_{max}, nm(ε)] in EtOH: 440 (51,100); in $CHCl_3$: 443 (60,700); in hexane: 438 (45,600). Mass spectrum, m/z: 227 (M^+). PMR spectrum ($CDCl_3$, δ, ppm): 6.6-7.65 (7H, H_β, H_γ, H_δ, Ph) and 3.0 and 3.14 (6H, NMe_2).

B) Similarly, 0.55 g (40%) of IIIb, with mp 204-205°C (from MeOH), was obtained from 0.86 g of indandione and 1.0 g of Ib in 10 ml of dry benzene. Found: C 74.5; H 6.34; N 5.72%. $C_{30}H_{30}N_2O_4$. Calculated: C 74.7; 6.23; N 5.81%. Electronic spectrum [λ_{max}, nm(ε)] in EtOH: 248 (63,300), 342 (73,600), 561 (125,000); in $CHCl_3$: 250 (33,500), 325 (33,700), and 578 (120,000).

After separation of IIIb, preparative TLC of the evaporated mother liquor on SiO_2 gave 0.36 g (25%) of IIb with mp 200-202°C (from MeOH). Found: C 74.71; H 6.58; N 5.83%. $C_{15}H_{15}NO_2$. Calculated: C 74.7; 6.23; N 5.81%. UV spectrum [λ_{max}, nm(ε)] in EtOH: 440 (81,000); in $CHCl_3$: 440 (68,000), 430 (35,000), and 460 (27,000). Mass spectrum, m/z: 241 (M^+). PMR spectrum ($CDCl_3$, δ, ppm): 6.84-7.1 (6H, H_β, H_δ, Ph), 2.32 (3H, Me), and 3.24 (6H, NMe). A 1-ml sample of a 10% solution of KOH in MeOH was added to 0.04 g of IIb in 3 ml of MeOH, after which the mixture was heated at 40°C for 10 min and evaporated to give XIIb, which was washed with MeOH. UV spectrum (λ_{max}, nm) in EtOH: 395. PMR spectrum (CD_3OD, δ, ppm): 2.18 (Me), 7.39 (CH), 7.46 (Ph), and 9.21 (CHO).

C) A mixture of 0.23 g of indandione and 0.3 g of Ic in 10 ml of absolute benzene was maintained at 20°C for 3 h, after which it was evaporated, and the residue was washed with absolute ether and recrystallized from acetone to give 0.17 g (42%) of IIc with mp 140-143°C. Electronic spectrum [λ_{max}, nm(ε)] in $CHCl_3$: 480 (38,836); in MeOH: 470 (16,484) and 450 (15,248); in hexane: 478 (30,198). PMR spectrum ($CDCl_3$, δ, ppm): 3.38 (6H, NMe_2), and 7.43-7.80 (6H, Ph, H_β and H_δ).

Potassium Salts IVa and IVb. A 0.5-ml sample of a 10% methanol solution of KOH was added to 0.05 g of IIIa in 10 ml of MeOH, and the mixture was refluxed for 10 min, during which a band with λ_{max} 285 nm (Va) appeared in the electronic spectrum instead of a band with λ_{max} 312 nm. After cooling, 0.04 g of slightly soluble IVa was separated and washed with MeOH. Electronic spectrum [λ_{max}, nm(ε)] in EtOH: 250 (40,000) and 560 (86,500). Salt IVb was similarly obtained. Electronic spectrum [λ_{max}, nm(ε)] in EtOH: 250 (36,300) and 561 (102,000). Salts IVa and IVb melted above 250°C.

Trimethylidyneoxanines VIa and VIb. A 2-ml sample of a 10% ethanol solution of HCl was added to 0.2 g of IIIa in 5 ml of EtOH, after which 0.15 g of VIa was separated in the form of a slightly soluble cherry-red precipitate, which was washed with H_2O and EtOH and had mp 235-237°C. Electronic spectrum [λ_{max}, nm(ε)] in EtOH: 250 (35,000) and 560 (87,600). Mass spectrum, m/z: 328 (M^+). Compound VIb, with mp 192-193°C, was similarly obtained. Electronic spectrum [λ_{max}, nm(ε)] in EtOH: 248 (29,400) and 561 (78,400). Mass spectrum, m/z: 342 (M^+). Found: C 77.22; H 4.41%. $C_{22}H_{14}O_4$. Calculated C 77.18; H 4.12%.

Condensation of Indandione with Aminals Id-f. A solution of 0.3 g (0.152 mole) of Id in 1 ml of absolute benzene was added to a warm solution of 0.18 g (0.123 mole) of indandione in 4 ml of absolute benzene, and the copious precipitate that formed immediately was washed with ether. Electronic spectrum of the precipitate (λ_{max}. nm) in EtOH: 250, 418, 540, and 665. Recrystallization from MeOH gave 0.15 g (48%) of IId in the form of a dark-violet precipitate with mp 227-229°C. Found: C 76.04; H 5.96%. $C_{16}H_{15}NO_2$. Calculated: C 75.8; H 5.93%. Electronic spectrum [λ_{max}, nm(ε)] in EtOH: 540 (144,000) and 250 (20,000). Mass spectrum, m/z: 253 (M^+). PMR spectrum (d_6-DMSO, δ, ppm): 3.07 and 3.24 (NMe_2), 5.97 (H_ε), 7.72 (H_β), 7.24-7.51 (3H, H_γ, H_δ, H_ξ), and 7.57 m (Ph); $J_{\varepsilon,\delta} = J_{\varepsilon,\xi} = J_{\beta,\gamma} = 12$ Hz.

A 2-ml sample of a 10% solution of KOH in MeOH was added to the mother liquor obtained after separation of the bulk of the IId, and the resulting precipitate was separated and washed with MeOH to give 0.07 g (29%) of IVd in the form of a dark-gray precipitate with mp > 250°C. Electronic spectrum [λ_{max}, nm(ε)] in EtOH: 245 (31,900), 665 (88,400), and shoulder at 560 (31,900). PMR spectrum (d_6-DMSO, δ, ppm): 7.27-7.83 m. The UV spectrum of the mother liquor after separation of IVd contained a band with λ_{max} 380 nm from aldehyde Vd and a weak band with λ_{max} 460 nm from aldehyde XIIa, which was formed from IId.

A 1-ml sample of a 10% solution of KOH in MeOH was added to a suspension of 0.2 g of IId in 5 ml of MeOH, after which the mixture was heated at 40°C for 10 min and cooled. The

0.2 g of XIIIa that separated was recrystallized from MeOH to give a product with mp > 300°C. Electronic spectrum (λ_{max}, nm) in EtOH: 460 (ε 57,598). PMR spectrum (CD_3OD, δ, ppm): 5.97 (H_α), 7.45 (h_β), 7.31 (H_γ), 7.1 (H_δ), 9.27 (CHO), and 7.4 (Ph); $J_{H_\alpha,CHO}$ = 9, $J_{\alpha,\beta}$ = 13.5, $J_{\beta,\gamma}$ = 12, and $J_{\gamma,\delta}$ = 15 Hz. A 0.1-g sample of XIIa was dissolved in 2 ml of MeOH, the solution was added to a solution of 0.2 g of $Me_2NH \cdot HCl$, and the mixture was heated at 45°C for 10 min. It was then cooled, and 0.07 g of IId was separated.

B) A 0.36-g sample of indandione was added to 0.5 g of Id, and the resulting doughy precipitate was washed with absolute ether three times and dissolved in 5 ml of absolute MeOH. A few drops of hydrochloric acid were added to the solution, and the resulting precipitate was separated and washed with water and MeOH to give 0.25 g (57%) of VId with mp 170-172°C. Found: C 77.66; H 3.91%. $C_{23}H_{14}O_4$. Calculated: C 77.96; H 3.98%. Electronic spectrum (λ_{max}, nm) in EtOH + 1% DMSO: 665 (ε 44,250) and 555 sh (ε 14,000). Mass spectrum, m/z: 354 (M^+). PMR spectrum (d_6-DMSO, δ, ppm): 7.0-8.15 m.

C) A solution of 0.6 g of Ie in 12 ml of dry benzene was added to 0.36 g of indandione in 10 ml of dry benzene. After 1 h, 0.6 g of IIe was separated and recrystallized from EtOH—$MeNO_2$ (1:2) to give a product with mp 176-177°C. Electronic spectrum (λ_{max}, nm) in EtOH: 630 (ε 101,388). Mass spectrum, m/z: 279 (M^+). PMR spectrum (d_6-DMSO, δ, ppm): 3.4 (NMe_2), 7.6 (H_β), 6.28 (H_ε), 5.88 (H_η), and 7.18-7.75 (8H, H_γ, H_δ, H_ξ, H_θ, and Ph).

D) A solution of 0.3 g of If in 25 ml of benzene was added to 0.18 g of indandione in 4 ml of dry benzene. After 1 h, 0.4 g of a slightly soluble almost black precipitate of IIf was separated and washed thoroughly with MeOH to give a product with mp 185-188°C. Found: N 4.1%. $C_{20}H_{19}NO_2$. Calculated: N 4.58%. Electronic spectrum (λ_{max}, nm) in DMSO—EtOH (1:6): 745 (ε 47,200). Mass spectrum, m/z: 305 (M^+).

Action of Aminal Ia on Methylidyneoxanne X. A 0.5-g sample of Ia was added to a suspension of 0.9 g of X in 10 ml of ether. After 24 h, the red precipitate was separated and recrystallized from MeOH. The precipitate was identified as X. Workup of the mother liquor gave 0.1 g of XI in the form of a yellow precipitate with mp 251-253°C. Electronic spectrum [λ_{max}, nm(ε)] in EtOH: 240 (87,500), 312 (60,000), and 470 (116,000). Potassium salt XIa, with mp > 250°C, was obtained from XI as described above.

CONCLUSIONS

Conjugated ω-dimethylamino β-diketones with two to five double bonds and trimethylidyne- and pentamethylidyneoxanine salts are formed in the condensation of aminals of conjugated β-dimethylamino aldehydes with indandione.

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