Association constant (K) values of the CT complexes studied were determined by using the Scott-modified (*10*), Benesi-Hildebrand (*11*) equation for a 1:1 complex

$$\frac{[A]\,[D]/}{d} = \frac{1}{K\epsilon} + \frac{[D]}{\epsilon}$$

where [A] and [D] are the initial molar concentration of the electron acceptor and donor, respectively, l is the optical path length of the cell, d is the optical density of the complex, K is the association constant (L·mol^{-1}), and ϵ is the molar extinction coefficient (L·mol^{-1}·cm^{-1}).

The wavelengths of maximum absorption, λ_{max}, molar extinction coefficients, ϵ, association constants, K, and transition energies, E, of the charge-transfer complexes of [2.2]isoindolinophanes **8a–f** with π acceptors are listed in Table V.

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Registry No. **2**, 102419-27-2; **4**, 102343-34-0; **6**, 102419-26-1; **7a**, 102343-41-9; **7b**, 102343-42-0; **8a**, 102343-36-2; **8a**·TCNE, 102343-43-1; **8b**, 102343-35-1; **8b**·TCNE, 102343-44-2; **8b**·CNIND, 102343-50-0; **8c**, 102343-37-3; **8c**·TCNE, 102343-45-3; **8c**·CNIND, 102343-51-1; **8c**·CHL, 102343-55-5; **8d**, 102343-38-4; **8d**·TCNE, 102343-46-4; **8d**·CNIND, 102343-52-2; **8d**·CHL, 102343-56-6; **8e**, 102343-39-5; **8e**·TCNE, 102343-47-5; **8e**·CNIND, 102343-53-3; **8e**·CHL, 102343-57-7; **8f**, 102343-40-8; **8f**·TCNE, 102343-48-6; **8f**·CNIND, 102343-54-4; **8f**·CHL, 102343-58-8; **9**, 102419-28-3; p-$ClC_6H_4NH_2$, 106-47-8; $PhNH_2$, 62-53-3; o-$CH_3C_6H_4NH_2$, 95-53-4; m-$CH_3C_6H_4NH_2$, 108-44-1; p-$CH_3C_6H_4NH_2$, 106-49-0; p-$CH_3OC_6H_4NH_2$, 104-94-9; CH_3NH_2, 74-89-5; $PhCH_2NH_2$, 100-46-9; p-toluenesulfonamide, 70-55-3; methanesulfonamide, 3144-09-0.

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The Stobbe Condensation. 5. Reactions of Aryl Aldehydes with α,α-Disubstituted Succinic Esters

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The condensation of some aryl aldehydes (Ia–i) with dimethyl 2,2-dimethylsuccinate gave predominantly the corresponding (E)-half-esters (IIIa–i). Hydrolysis of III produced the dibasic acids (V), which were dehydrated to the anhydrides (VI). These were converted to the acids VIII. Cyclization of III revealed the formation of VII or IX, depending on reaction conditions.

The Stobbe condensation of α,α-disubstituted succinic esters with carbonyl compounds has not been thoroughly investigated (*1*, *2*). Thus, dimethyl 2,2-dimethylsuccinate was condensed with benzaldehyde, o-chloro-, m-chloro-, o-methoxy-, and p-methoxybenzaldehydes and 1-naphthaldehyde, as well as thiophene- and 1-methylpyrrole-2-carboxaldehydes (Ia–i), to form almost exclusively the (E)-half-esters (III) (cf. Scheme I).

The structure of the half-esters (III) was evident from their spectral data (*3a*,*b*; *4*) and chemical behavior (cf. Table I and II). Saponification of the half-esters afforded the acids (Vb–i), which were converted to the anhydrides (VIb–i). These anhydrides were also obtained from the half-esters upon treatment with sodium acetate–acetic anhydride mixtures (*5*).

The (E) configuration of the half-esters (III) is established by their cyclization under mild conditions (*7*) to the ketonic esters (VII) (cf. Scheme II). It should also be noted that the precursors of these (E)-half-esters, the δ-lactones, are free from steric and polar interactions (*6*).

Table I. Infrared and Mass Spectra of Some Representatives of Compounds III–IX

compd	infrared spectra(KBr) ν, cm^{-1}	mass spectra m/e	% of base peak	assign.
IIIc	1710 (s) } C=O	278	4.44	$M^{\bullet +}$
	1720 (s) }	174	100	$[C_{12}H_{14}O]^+$
		159	41.76	$[C_{11}H_{11}O]^+$
	2980 (br) OH	151	38.38	$[C_{10}H_{15}O]^+$
Vc	1685 (s) } C=O			
	1710 (s) }			
	2980 (br) OH			
VIc	1775 (s) } C=O			
	1820 (s) }			
VIIg	1710 (s) } C=O	265	8.44	$M^{\bullet +}$
		170	92.06	$[C_{12}H_{10}O]^+$
	1745 (s) }	142	100	$[C_{10}H_6O]^+$
		141	92.47	$[C_{10}H_5O]^+$
VIIIc	1702 (s) } C=O	246	25.67	$M^{\bullet +}$
	1710 (s) }	201	100	$[M\text{-}CO_2H]^+$
	2990 (br) OH			
IXc	1704 (s) } C=O	246	94.88	$M^{\bullet +}$
	1775 (s) }	159	100	$[C_{10}H_7O_2]^+$

Treatment of the anhydrides (VI) with aluminum chloride and cyclization of the produced oxoindenyl acids (VIII) gave the ketonic lactones (IX). These were also obtained by treatment of half-esters (III) with concentrated sulfuric acid under more drastic conditions (*7–9*), to affect isomerization of the inter-

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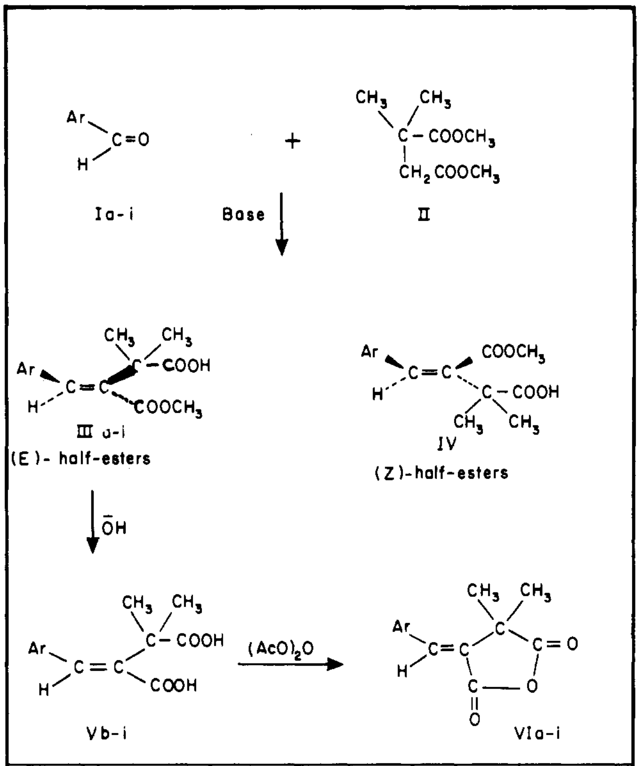
Table II. Melting Points, Yields, and Electronic and Nuclear Magnetic Resonance Spectral Data of Compounds III and V–IX

compd	mp, °C	yield, %	electronic spectra (ethanol)		NMR ($CDCl_3$) σ (no. of protons)
			λ_{max}, nm	ϵ_{max}	
IIIa	109	95	228	11620	11.73 (s, 1)
					7.53–7.06 (m, 4)
					3.86 (s, 3)
					1.5 (s, 6)
IIIb	149–150	94	378	23910	9.06 (s, 1)
					7.53–6.03 (m, 4)
			295–300	4000	3.75 (s, 3)
					3.6 (s, 3)
					1.41 (s, 6)
IIIc	94	94	269	11175	11.43 (s, 1)
					7.7 (d, 2)
					7.10 (d, 2)
					3.78 (s, 3)
					3.61 (s, 3)
					1.48 (s, 6)
IIId	91	96	296	4710	11.40 (s, 1)
			258	7630	7.46–6.93 (m, 5)
					3.86 (s, 3)
					3.60 (s, 3)
					1.53 (s, 6)
IIIe	110	95	293	9105	12.26 (s, 1)
			225	52150	8.06–7.5 (m, 8)
					3.40 (s, 3)
					1.66 (s, 6)
IIIf	116	97	250	8875	11.43 (s, 1)
					7.06–7.03 (m, 5)
					3.5 (s, 3)
					1.53 (s, 6)
IIIg	114	96	256	13510	11.93 (s, 1)
					8.06–7.13 (m, 4)
					6.78 (s, 1)
					3.58 (s, 3)
					1.63 (s, 6)
IIIh	135	97	259	16590	8 (d, 2)
					7.42 (d, 2)
					6.79 (s, 1)
					3.89 (s, 3)
					1.49 (s, 6)
IIIi	134	98	255	11550	11.16 (s, 1)
					7.25 (s, 5)
					6.85 (s, 1)
					3.56 (s, 3)
					1.52 (s, 6)
Vb	149–150	95	372	3715	7.06 (s, 1)
			305–295	1150	6.93 (s, 2)
			227	6455	6.53–6.13 (m, 3)
					3.9 (s, 3)
					1.53 (s, 6)
Vc	134–135	93	270	16830	10.4 (s, 2)
					8.32 (d, 2)
					7.3 (d, 2)
					7.53 (s, 1)
					4.06 (s, 3)
					1.73 (s, 6)
Vd	138–139	92	295	3700	9.86 (s, 2)
			257	6660	7.8–7.2 (m, 5)
					4.00 (s, 3)
					1.6 (s, 6)
Ve	159–160	95	290	10785	8.02–7.48 (m, 7)
			225	115040	7.4 (s, 1)
					6.46 (s, 2)
					1.72 (s, 6)
Vf	144–146	94	254	8395	9.00 (s, 2)
					7.53–7.47 (m, 4)
					7.13 (s, 1)
					1.56 (s, 6)
Vg	140	93	256	4860	8.00 (s, 2)
					7.52–7.28 (m, 4)
					6.64 (s, 1)
					1.48 (s, 6)
Vh	126	95	265	23365	9.9 (s, 2)
					7.23 (s, 4)
					6.88 (s, 1)
					1.53 (s, 6)
Vi	159	95	258	11965	7.73 (s, 5)
					7.03 (s, 1)
					6.27 (s, 2)
					1.53 (s, 6)
VIa	99	92	338	15270	8.00–7.30 (m, 4)
			290 (sh)	4825	1.56 (s, 6)
VIb	98	93	337	12825	7.93 (s, 1)
			227	3130	7.20–6.46 (m, 3)
					3.93 (s, 3)
					1.73 (s, 6)
VIc	105	94	335	24600	8.06 (d, 2)
			233	12450	7.73 (d, 2)
					7.53 (s, 1)
					3.46 (s, 3)
					1.7 (s, 6)
VId	87	95	335–340	5940	7.38–6.8 (m, 5)
			300	6515	
			239	5745	3.87 (s, 3)
					1.53 (s, 6)
VIe	125	99	325	6080	8.4–7.8 (m, 7)
			221	63255	7.67 (s, 1)
					1.8 (s, 6)
VIf	100	93	290–295	3570	7.37–7.15 (m, 5)
			263	3385	1.5 (s, 6)
VIg	104	92	292	11720	7.88–7.28 (m, 4)
			225	9340	6.92 (s, 1)
					1.56 (s, 6)
VIh	137	95	306	13835	7.88 (d, 2)
			228	7340	7.40 (d, 2)
					6.92 (s, 1)
					1.52 (s, 6)
VIi	81	94	302	12960	7.33 (s, 5)
			226	3455	7.16 (s, 1)
			225	3435	1.53 (s, 6)
VIIe	91	45	335	920	7.90–7.36 (m, 7)
			288 (sh)	18420	3.64 (s, 3)
			277	28090	
			270	30395	1.50 (s, 6)
			227	44900	
VIIf	80	30	300	1695	7.4–7.12 (m, 4)
			247	18665	3.67 (s, 3)
			241	15840	1.48 (s, 6)
VIIg	65	50	325	2320	7.28–6.98 (m, 4)
			244	55730	3.67 (s, 3)
					1.47 (s, 6)
VIIIc	144–145	75	290	970	7.73 (s, 1)
			251	30510	7.20–6.7 (m, 4)
					3.77 (s, 3)
					1.48 (s, 6)
VIIIf	203–205	73	328	920	7.67–7.4 (m, 4)
			248	47430	5.13 (s, 1)
			241	46970	1.53 (s, 6)
VIIIg	164–165	75	325	290	8.67 (s, 1)
			245	19425	
			242	19135	7.47–7.2 (m, 3)
					7.00 (s, 1)
					1.53 (s, 6)
VIIIh	215	74	280	1170	7.48–7.23 (m, 3)
			248	43310	7.03 (s, 1)
			241	39505	1.47 (s, 6)
VIIIi	182–184	73	315	465	7.43 (s, 4)
			244	40130	7.33 (s, 1)
			238	34285	7.00 (s, 1)
					1.53 (s, 1)
IXc	114	40	315	1310	7.72–7.18 (m, 3)
			252	3420	5.85 (d, 1)
			223	15520	3.85 (s, 3)
					3.25 (d, 1)
					1.52 (s, 3)
					1.18 (s, 3)
IXe	105	30	299 (sh)	4330	8.07–7.6 (m, 6)
			285	6235	6.32 (d, 1)
			277 (sh)	5540	3.42 (d, 1)
			251	38100	1.26 (s, 3)

Table II (Continued)

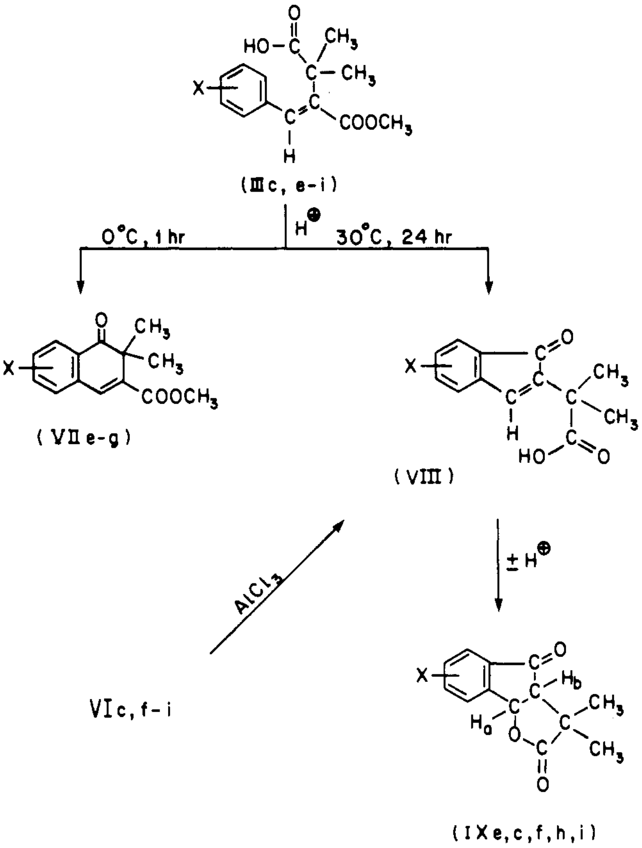
compd	mp, °C	yield, %	electronic spectra (ethanol) λ_{max}, nm	electronic spectra (ethanol) ϵ_{max}	NMR ($CDCl_3$) σ (no. of protons)
IXf	101	40	276	1820	7.72–7.4 (m, 3)
					6.04 (d, 1)
			252	4340	3.35 (d, 1)
			246	3900	1.54 (s, 3)
					1.26 (s, 3)
IXh	100	30	300	1370	7.7 (s, 3)
			293	1425	5.87 (d, 1)
			246	9010	3.28 (d, 1)
					1.52 (s, 3)
					1.17 (s, 3)
IXi	78	50	278	1830	7.68 (s, 4)
			241	10985	5.88 (d, 1)
					3.25 (d, 1)
					1.50 (s, 3)
					1.17 (s, 3)

Scheme I



Compound	Ar	Compound	Ar	Compound	Ar
I–VI a	C_4H_3S	d	o-OCH_3-C_6H_4	g	m-Cl-C_6H_4
b	C_5H_6N	e	-$C_{10}H_8$	h	p-Cl-C_6H_4
c	p-OCH_3-C_6H_4	f	o-Cl-C_6H_4	i	C_6H_5

Scheme II



mediate acylium cation (cf. Scheme II).

The structure of all the above products was substantiated by chemical and spectral analyses (cf. Table I and II).

Experimental Section

Microanalyses were performed by Prof. Malissa and G. Reuter, Analytisches Laboratorium BRD, and are provided for review. Infrared spectra (KBr disk or carbon tetrachloride) were measured on Perkin-Elmer 520B. Nuclear magnetic resonance spectra were recorded for solutions in deuteriochloroform with tetramethylsilane as external standard on Varian T60A and Jeol JNM-MH 100 spectrometers. Electronic spectra were taken for solutions in ethyl alcohol on Pye Unicam Sp 8000 recording spectrometer. The mass spectra were measured with Varian MAT 311A spectrometer. Melting points were determined by using a Bock-Monoscop M (thermal microscope).

Preparation of the Half-Esters (IIIa-i). General Procedure. (a) Sodium Hydride Method. The dimethyl 2,2-dimethylsuccinate (0.12 mol), aldehyde (0.1 mol), and sodium hydride (0.15 mol) are stirred in excess dry benzene and then worked up as previously reported (*10*). The residue was crystallized from *n*-hexane or cyclohexane to produce the crystalline (*E*)-half-esters (III).

(b) Potassium tert-Butoxide Method. The dimethyl 2,2-dimethylsuccinate (0.06 mol) and aldehyde (0.05 mol) in absolute *tert*-butyl alcohol are added to *tert*-butoxide (from 3 g metallic potassium in 50 mL absolute *tert*-butyl alcohol) within 15 min and then worked up as previously reported (*10*). The crude product was crystallized as before to give (*E*)-3-methoxycarbonyl-2,2-dimethyl-4-(aryl)-but-3-enoic acids (IIIa–i).

Saponification of the Half-Esters (IIIa-i) to the Diacids (Vb-i). The half-ester (2 g) was refluxed with 15% aqueous alcoholic potassium hydroxide solution (15 g KOH, 50 mL H_2O, and 50 mL methanol) for 4 h and worked up as before (*10*). Crystallization of the acids (Vb–i) with *n*-hexane gave (*E*)-3-

carboxy-2,2-dimethyl-4-(aryl)-but-3-enoic acids (Vb–i) while (IIIa) produced (VIa) directly.

Conversion of the Dibasic Acids to the Corresponding Anhydrides (VIa–i). Acetic anhydride (10 mL) was added to the dibasic acid (1 g), refluxed for 5 h, and worked up as previously reported (*10*). The crude products were crystallized from hexane to give (*E*)-3-carboxy-2,2-dimethyl-4-(aryl)-3-enoic anhydrides (VIa–i).

Treatment of Half-Esters (IIb,c) with Acetic Anhydride and Sodium Acetate. A mixture of the half-ester (1 mol), freshly fused sodium acetate (2 mol), and excess of acetic acid and acetic anhydride was refluxed for 10–12 h and worked up as before (*10*). The products were crystallized from cyclohexane to produce the corresponding anhydrides (VIb,e).

Treatment of the Half-Ester (IIIa,c,e–i) with Concentrated Sulfuric Acid. The half-esters (IIIe–g) were added in portions to cold concentrated sulfuric acid (10 mL per 0.5 g) and left in ice for a period of 1 h. The mixture was poured into ice-cold water and the product was extracted with ether. The ethereal layer was washed with 5% sodium carbonate solution and then with cold water and finally dried (Na_2SO_4). Ether was evaporated, and the organic product was crystallized from petroleum ether (40–60 °C) or hexane to give methyl 3,4-dihydro-3,3-dimethyl-4-oxophenanthrene-2-carboxylate (VIIe), methyl 5-chloro-1,2-dihydro-2,2-dimethyl-1-oxonaphthalene-3-carboxylate (VIIf), and methyl 6-chloro-1,2-dihydro-2,2-dimethyl-1-oxonaphthalene-3-carboxylate (VIIg).

When the half-esters (IIIc,e,f,h,i) were treated with concentrated sulfuric acid and left overnight at room temperature, they produced 3,3-dimethyl-2-oxotetrahydrofurano-(4,5b)-6-methoxyindan-4-one (IXc), -(1,2d)-benzo[*e*]indan-3-one (IXe), -(4,5b)-8-chloroindan-4-one (IXf), -(4,5b)-6-chloroindan-4-one (IXh), and -(4,5b)-indan-4-one (IXi).

Treatment of the Anhydrides (VIc,f–i) with Anhydrous Aluminum Chloride. To a stirred solution of the anhydride in *sym*-tetrachloroethane (10 mL per 1 g anhydride), an excess amount of anhydrous aluminum chloride was added in one portion and stirring was continued for 6 h and the mixture was left overnight at room temperature. The material was poured into a mixture of ice and concentrated hydrochloric acid and the acidic product was extracted with ether. The ethereal layer was extracted with 10% carbonate solution, and the carbonate layer was acidified with cold dilute hydrochloric acid. The precipitated oxoindenyl acids (VIIIc,f–i) were extracted with ether, washed with cold water, dried (using Na_2SO_4), and filtered and the ether was distilled off. The oxoindenyl acids (VIIIc,f–i) obtained were crystallized from petroleum ether (bp 40–60 °C) to give 2-(6-methoxy-1-oxo-2-indenyl)- (VIIIc), 2-(4-chloro-1-oxo-2-indenyl)- (VIIIf), 2-(5-chloro -1-oxo-2-indenyl)- (VIIIg), 2-(6-chloro-1-oxo-2-indenyl)-2-methylpropanoic acids (VIIIh), and 2-methyl-2-(1-oxo-2-indenyl)-propanoic acid (VIIIi).

Cyclization of the Oxoindenyl Acids (VIIIc,f,h,i) with Concentrated Sulfuric Acid. The oxoindenyl acid was added in portions to cold concentrated sulfuric acid (10 ml per 0.5 g) and left overnight at room temperature. The mixture was poured into ice-cold water and the product was extracted with ether. The ethereal layer was washed with 5% sodium carbonate solutions and then cold water and finally dried (Na_2SO_4). Ether was evaporated and the remaining material was crystallized from petroleum ether (bp 40–60 °C) to give the ketonic lactones (IXc,f,h,i).

Registry No. Ia, 98-03-3; Ib, 1192-58-1; Ic, 123-11-5; Id, 135-02-4; Ie, 66-77-3; If, 89-98-5; Ig, 587-04-2; Ih, 104-88-1; Ii, 100-52-7; II, 49827-44-3; IIIa, 102696-75-3; IIIb, 102696-76-4; IIIc, 102696-77-5; IIId, 102696-78-6; IIIe, 102696-79-7; IIIf, 102696-80-0; IIIg, 102696-81-1; IIIh, 102696-82-2; IIIi, 102696-83-3; Vb, 102696-84-4; Vc, 102696-85-5; Vd, 102779-85-1; Ve, 102696-86-6; Vf, 102696-87-7; Vg, 102696-88-8; Vh, 102724-22-1; Vi, 102724-23-2; VIa, 102696-89-9; VIb, 102696-90-2; VIc, 102696-91-3; VId, 102696-92-4; VIe, 102696-93-5; VIf, 102696-94-6; VIa, 102696-95-7; VIh, 102696-96-8; VIi, 102696-97-9; VIIe, 102696-98-0; VIIf, 102696-99-1; VIIg, 102697-00-7; VIIIc, 102697-01-8; VIIIf, 102697-02-9; VIIIg, 102697-03-0; VIIIh, 102724-24-3; VIIIi, 102697-04-1; IXc, 102724-25-4; IXe, 102697-05-2; IXf, 102697-06-3; IXh, 102697-07-4; IXi, 102697-08-5.

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Polynitroalkyl Ethers by Direct Nitroalkylation

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The synthesis of symmetrical 3,3-dinitro and 3,3,3-trinitroalkyl ethers and of unsymmetrical ethers with one 3,3-dinitro or 3,3,3-trinitroalkyl group and one 2,2-dinitroalkyl group was accomplished by the reaction of the alcohols with triflic acid.

We were interested in synthesizing polynitro-substituted ethers such as **2** for a study of their properties such as melting point. There was no existing method for easily synthesizing such compounds. Although ethers with electron-withdrawing groups, such as nitro, in only one of the alkyl groups are readily accessible by the alkylation of the substituted alcohol (*1*), ethers with two polynitroalkyl groups have had to be synthesized indirectly (*2*). This usually involves alkylation of the polynitro alcohol with a group that can then be converted to a polynitro

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