on AEI (UK) mass spectrophotometer Model No. MS 3074 at 70 eV.

Acid Chlorides. The acid chlorides were prepared by the action of thionyl chloride on the corresponding carboxylic acids and then vacuum distilled.

Hydroxylamines. *N*-phenyl- and *N*-4-chlorophenylhydroxylamines were freshly prepared by the reduction of nitrobenzene and 4-chloronitrobenzene, respectively, with zinc dust and ammonium chloride in aqueous alcoholic solutions (*8*).

Hydroxamic Acids. The procedure for the preparations are essentially the same as that reported earlier (*8*). The acids are synthesized by reacting the arylhydroxylamine with acid chloride at low temperature in diethyl ether containing aqueous suspensions of sodium bicarbonate.

Colorimetric Tests. The well-known color reaction which takes place in 6 M HCl between vanadium(V) and hydroxamic acid was adopted (*1–3*). The standard vanadium was extracted with a known volume of hydroxamic acid solution (in chloroform). The violet-colored layer was separated and diluted to 25 mL with chloroform, and the absorbance was measured against a reagent blank. The concentration of the hydroxamic acid was computed from a standard calibration curve.

Potentiometric Tests. A known weight of the hydroxamic acid is dissolved in 50 mL of dimethylformamide and titrated with 0.1 M sodium methoxide by using platinum and calomel electrode (saturated KCl in methanol) in the presence of nitrogen.

Ionization Constants. These were determined at 35 ± 0.1 °C in 70% dioxan water medium (Table II). The calculation method for pK_a is described elsewhere (*1*).

The ionization of hydroxamic acid (HA) in aqueous solution gives hydrogen ion and hydroxamate ion and the equilibrium constant may be

$$K_a(\mathrm{aq}) = ([\mathrm{H}^+][\mathrm{A}^-])/[\mathrm{HA}]\,Y_{\mathrm{H}^+}Y_{\mathrm{A}^-}/Y_{\mathrm{HA}} \qquad \text{(i)}$$

or

$$K_a = -\log [\mathrm{H}^+] + \log [\mathrm{HA}]/[\mathrm{A}^-] - 2 \log Y_\pm \qquad \text{(ii)}$$

assuming the activity coefficient of un-ionized acid is unity and the empirical correction for "medium effect" as suggested by Van Uitert and Haas (*1*, *9*) as

$$-\log [\mathrm{H}^+] = B + \log U_{\mathrm{H}}{}^0 + \log Y_\pm \qquad \text{(iii)}$$

The final form of the equation for calculating the ionization constants in dioxan–water mixture is

$$pK_a = B + \log U_{\mathrm{H}^0} + \log [\mathrm{HA}]/[\mathrm{A}^-] - \log Y_\pm \qquad \text{(iv)}$$

where B is the pH meter reading and $\log U_{\mathrm{H}^0}$ is determined experimentally (*1*, *10*).

In the thermostated (35 ± 0.1 °C) titration vessel carrying a glass electrode, a calomel electrode, and a microburet (5 × 0.01 mL), 0.25 M of hydroxamic acid and 47.5 mL of solvent (70% v/v dioxan:water) were taken. It was titrated after being deaerated by passage of nitrogen (presaturated with solvent), and after 15 min the highest steady B value was noted after each increment.

Registry No. I, 91097-78-8; II, 95333-99-6; III, 95334-00-2; IV, 97877-85-5; V, 97877-86-6; *p*-$BuOC_6H_4C(O)Cl$, 33863-86-4; *p*-$ClC_6H_4OCH_2CH(CH_3)C(O)Cl$, 97877-84-4; *p*-$ClC_6H_4NHOH$, 823-86-9; PhNHOH, 100-65-2; 3,4,5-trimethoxycinnamoyl chloride, 10263-19-1.

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Structural Information on Uranyl Sulfate Complexes with Some Bidentate Ligands

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Coordination compounds formed by uranyl sulfate with 1,10-phenanthroline, 2,2′-bipyridyl, 2,2′-bipyridyl *N*,*N*′-dioxide, 2,2′-bipyridylamine, 4,4′-bipyridyl, 4,4′-bipyridyl *N*,*N*′-dioxide, methylpyrazine, quinoxaline, 2,3-dimethylquinoxaline, hexamethylenetetramine, 1,3-di-4-pyridylpropane, 2-methylbenzothiazole, and nicotinic acid have been prepared. The compounds isolated in the solid state have been characterized by analytical data and IR spectroscopy down to 200 cm^{-1} to elucidate the U(VI)–ligand and U(VI)–sulfate bonding modes. Tentative coordination number and stereochemical environments around U(VI) are assigned in each case.

Introduction

Coordination compounds formed by transition- and nontransition-metal(II) salts with potentially bidentate ligands with nitrogen and/or oxygen/sulfur as the donor sites have been the subject of extensive investigations. Few studies have, however, appeared on the complexes of uranyl salts with such ligands. The present communication describes the coordination compounds formed by the interaction of uranyl sulfate with 1,10-phenanthroline (phen), 2,2′-bipyridyl (2,2′-bpy), 2,2′-bipyridyl *N*,*N*′-dioxide (2,2′-bpyO_2), 2,2′-bipyridylamine (2,2′-bpyA), 4,4′-bipyridyl (4,4′-bpy), 4,4′-bipyridyl *N*,*N*′-dioxide (4,4′-bpyO_2), methylpyrazine (MP), quinoxaline (Q), 2,3-dimethylquinoxaline (DMQ), hexamethylenetetramine (HMTA), 1,3-di-4-pyridylpropane (DPP), 2-methylbenzothiazole (MBT), and nicotinic acid (NA) (Figure 1). IR spectroscopy has been used to elucidate the U(VI)–ligand and U(VI)–sulfate bonding modes and the tentative stereochemistry around U(VI) in each case.

Experimental Section

Materials. Uranyl sulfate was obtained from B.D.H. and used as such. Phen, 2,2′-bpy, 4,4′-bpy, 2,2′-bpyA, Q, MBT, and NA

Table I. Infrared Spectral Data (cm^{-1})

compd	uranyl modes		sulfato modes			
	ν_{as}(UO)	δ(OUO)	ν_1	ν_2	ν_3	ν_4
$UO_2(phen)SO_4$	926	259	1000	466	1222, 1175, 1095	640, 620, 603
$UO_2(2,2'\text{-}bpy)SO_4$	937	258	1015	470	1215, 1134, 1070	645, 610, 578
$UO_2(2,2'\text{-}bpyO_2)SO_4$	925	262	972	465	1230, 1175, 1085	640, 625, 602
$UO_2(4,4'\text{-}bpy)SO_4$	930	257	970	470	1040, 1130, 590	655, 602, 590
$UO_2(2,2'\text{-}bpyA)_2SO_4$	912	245	970	458	1240, 1165, 1100, 1045	641, 620, 600
$UO_2(4,4'\text{-}bpyO_2)SO_4$	917	260	985	472	1234, 1100, 1066	640, 632, 602
$UO_2(MP)SO_4$	924	248	1010	480	1150–1040	640
$UO_2(Q)_2SO_4$	934	254	1010	470	1216, 1120, 1040	640, 632, 612
$UO_2(DMQ)_2SO_4$	940	252	1008	470	1210, 1145, 1050	650, 630, 610
$UO_2(HMTA)_2SO_4$	910	264	1015	470	1245, 1163, 1120	640, 613, 595
$UO_2(DPP)SO_4$	908	255	998	475	1249, 1166, 1108, 1040	627, 614
$UO_2(MBT)SO_4$	942	258	1004	460	1240, 1142, 1058	641, 616, 600
$UO_2(NA)_2SO_4$	911	251	1003	467	1256, 1100, 1035	616, 600

Table II. U(VI)-Ligand and U(VI)-Sulfate Bonding Modes and Tentative Coordination Number Around U(VI)

compd	mode of U(VI)–ligand bonding	ref	mode of U(VI)–sulfate bonding	tentative coordination no. of U(VI)
$UO_2(phen)SO_4$	bidentate chelating	9	bidentate chelating	six
$UO_2(2,2'\text{-}bpy)SO_4$	bidentate chelating	9	bidentate chelating	six
$UO_2(2,2'\text{-}bpyO_2)SO_4$	bidentate chelating	10	bidentate chelating	six
$UO_2(4,4'\text{-}bpy)SO_4$	bidentate bridging	11–13	bidentate chelating	six
$UO_2(4,4'\text{-}bpyO_2)SO_4$	bidentate bridging	14	bidentate chelating	six
$UO_2(2,2'\text{-}bpyA)_2SO_4$	bidentate chelating	15	bidentate chelating	eight
$UO_2(MP)SO_4$	terminal monodentate	16–22	tetradentate bridging	seven
$UO_2(Q)_2SO_4$	terminal monodentate	16–22	bidentate chelating	six
$UO_2(DMQ)_2SO_4$	terminal monodentate	16–22	bidentate chelating	six
$UO_2(HMTA)_2SO_4$	bidentate bridging	23–28	bidentate chelating	eight
$UO_2(DPP)SO_4$	bidentate bridging	29	bidentate chelating	six
$UO_2(MBT)SO_4$	terminal N-bonded monodentate	30, 31	bidentate chelating	five
$UO_2(NA)_2SO_4$	terminal N-bonded monodentate	30	bidentate chelating	six

(all from Koch-Light Laboratories, England), MP (Wyandotte Chemicals Corp., U.S.A.), DMQ (Sigma Chemicals Co., U.S.A.), HMTA (Dr. Theodor Schuchardt GmbH & Co., Munich), and DPP (Reilly Tar & Chemicals Corporation, U.S.A.) were used as such. $2,2'\text{-}bpyO_2$ and $4,4'\text{-}bpyO_2$ were obtained by oxidizing the respective bipyridyls (*1*).

Synthesis of the Complexes. The uranyl sulfate complexes were prepared by mixing together an excess of the ligand solution and the uranyl sulfate solution in methanol. The complexes which precipitated immediately or crystallized out on standing were filtered by suction, washed with methanol, and dried in an air oven.

Analysis of the Complexes. Uranium and sulfate estimations were submitted for review (*2*). Uranium was determined gravimetrically as oxinate by using the standard procedure after destroying the organic moiety with concentrated nitric acid and then with concentrated sulfuric acid. Sulfate was estimated gravimetrically as $BaSO_4$.

Infrared Spectra. IR spectra of the uncoordinated ligand and of the complexes were recorded as Nujol mulls (neat in the case of liquid ligands) supported between sodium chloride plates (rock salt region) and thin polyethylene sheets (650–200 cm^{-1}) on a Perkin-Elmer 621 spectrophotometer equipped with cesium iodide optics.

Results and Discussion

The uranyl sulfate complexes synthesized in the present study together with IR frequencies due to uranyl (*3*) and sulfates (*4–8*) groups are listed in Table I. The U(VI)–ligand and U(VI)–sulfate bonding modes and the tentative coordination number around U(VI) in each case are listed in Table II. Tentative stereochemical environments around U(VI) in some representative complexes are shown in Figure 2.

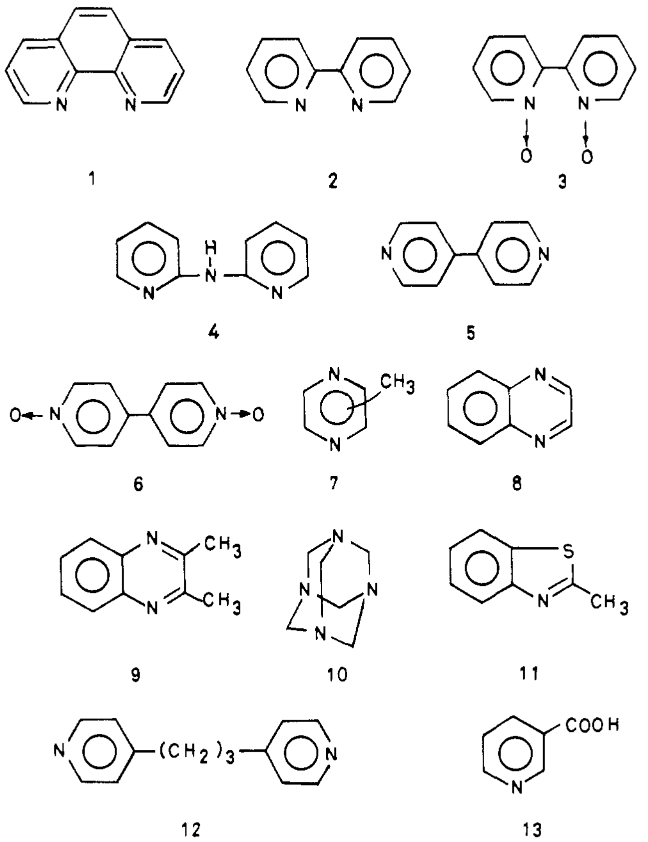

Figure 1. Ligand used: (1) 1,10-phenanthroline; (2) 2,2′-bipyridyl; (3) 2,2′-bipyridyl *N*,*N*′-dioxide; (4) 2,2′-bipyridylamine; (5) 4,4′-bipyridyl; (6) 4,4′-bipyridyl *N*,*N*′-dioxide; (7) methylpyrazine; (8) quinoxaline; (9) 2,3-dimethylquinoxaline; (10) hexamethylenetetramine; (11) 2-methylbenzothiazole; (12) 1,3-di-4-pyridylpropane; (13) nicotinic acid.

On the basis of considerations of U(VI)–ligand and U(VI)–sulfate bonding modes (Tables I, II) the tentative coordination numbers and the stereochemical environments for the uranyl sulfate complexes are suggested as follows: (i) $UO_2(phen)SO_4$ and $UO_2(2,2\text{-}'bpy)SO_4$ are monomeric structures involving 6-

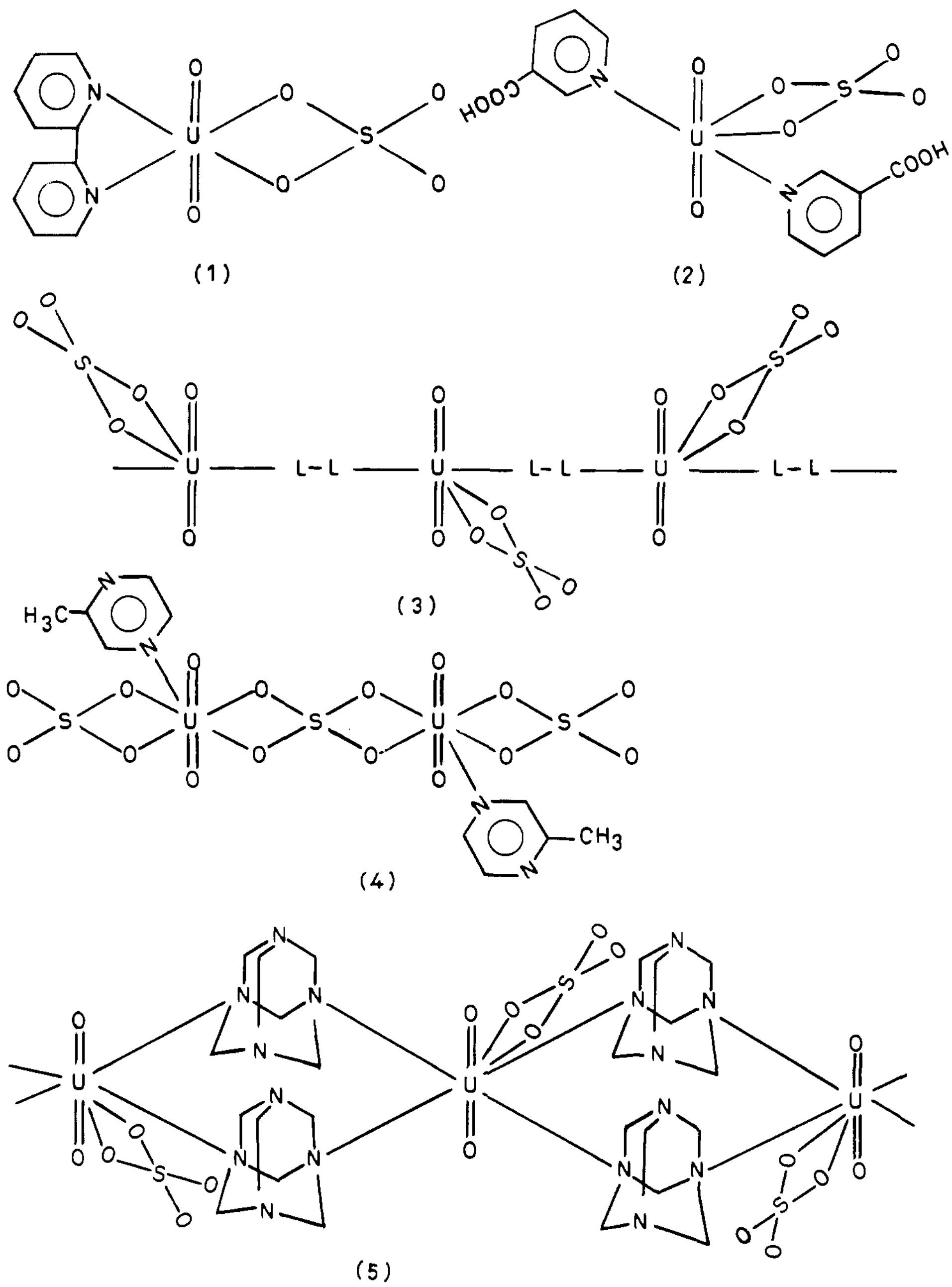

Figure 2. Tentative structures of (1) $UO_2(2,2'\text{-bpy})SO_4$; (2) $UO_2(NA)_2SO_4$; (3) $UO_2(LL)SO_4$ (LL = 4,4'-bpy, 4,4'-bpyO_2, DPP); (4) $UO_2(MP)SO_4$; (5) $UO_2(HMTA)_2SO_4$.

coordinated U(VI) with four ligand atoms (two nitrogens from phen and 2,2'-bpy molecules and two oxygen atoms from sulfate groups) forming a tetragonal equatorial ring around the linear OUO group; (ii) $UO_2(2,2'\text{-bpy}O_2)SO_4$ has a monomeric structure involving 6-coordinated U(VI) with four oxygen atoms (two from bpyO_2 molecule and two from sulfate group) forming a tetragonal ring around the linear uranyl group; (iii) the 1:2 uranyl sulfate complexes with Q, DMQ, and NA are monomeric structures involving 6-coordinated U(VI) with four ligand atoms—two nitrogens of the terminally bonded Q, DMQ, or pyridine ring nitrogen-bonded NA molecules and two oxygens of the sulfate ligand—forming a tetragonal equatorial ring around the linear OUO group; (iv) 1:1 uranyl sulfate complexes with 4,4'-bpy and DPP have polymeric chain structures involving 6-coordinated U(VI) with two nitrogens of the bidentate bridging 4,4'-bpy/DPP molecules and two oxygens of the sulfate groups forming a tetragonal ring around the linear uranyl group; (v) $UO_2(4,4'\text{-bpy}O_2)SO_4$ is a polymeric chain structure involving 6-coordinated U(VI) with four oxygen atoms (two from bridging 4,4'-bpyO_2 molecules and two from sulfate groups) in the equatorial plane around the linear OUO group; (vi) $UO_2(MBT)SO_4$ has a monomeric 5-coordinated trigonal-bipyramidal geometry with three atoms (one nitrogen from MBT and two oxygens of the sulfato ligand) forming an equatorial ring around the uranyl group; (vii) $UO_2(MP)SO_4$ is a linear polymeric chain structure involving 7-coordinated U(VI) with five ligand atoms (four oxygens of the tetradentate bridging sulfato groups and one nitrogen from the terminally bonded monodentate MP molecules) forming a planar pentagonal ring around the linear OUO group; (viii) $UO_2(2,2'\text{-bpyA})_2SO_4$ is assigned to have monomeric 8-coordinated U(VI) with six ligand atoms (four nitrogens from two 2,2'-bpyA molecules and two oxygens of the sulfato ligand) forming a hexagonal equatorial ring around the linear uranyl group; and (ix) the $UO_2(HMTA)_2SO_4$ complex is considered to have a linear polymeric chain structure having 8-coordinated U(VI) with six ligand atoms—four nitrogens from two bidentate bridging HMTA molecules retaining the chair configuration of the uncoordinated ligand (*32*) and two oxygens from bidentate chelating sulfato groups—around the linear OUO group.

Registry No. $UO_2(phen)SO_4$, 42565-88-8; $UO_2(2,2'\text{-bpy})SO_4$, 40415-75-6; $UO_2(2,2'\text{-bpyO}_2)SO_4$, 50989-29-2; $UO_2(4,4'\text{-bpy})SO_4$, 51024-41-0; $UO_2(2,2'\text{-bpyA})_2SO_4$, 63162-30-1; $UO_2(4,4'\text{-bpyO}_2)SO_4$, 96789-83-2; $UO_2(MP)SO_4$, 76772-31-1; $UO_2(Q)_2SO_4$, 73689-28-8; $UO_2(DMQ)_2SO_4$, 73700-17-1; $UO_2(HMTA)_2SO_4$, 71769-14-7; $UO_2(DPP)SO_4$, 60647-93-0; $UO_2(MBT)SO_4$, 68229-42-5; $UO_2(NA)_2SO_4$, 67799-02-4.

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Spectroscopic Study of Some New *N*-Aryl Pyrroles

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A series of new 2,5-dimethyl-N-substituted pyrroles (I) were obtained by condensation of 2,5-hexanedione with different amines. The UV data show that about 11.7% of the total electronic density of the nitrogen of pyrrole ring is conjugated with the aryl group. A correlation between the UV and NMR results has been established, and the mass spectra of these derivatives have also been measured and discussed.

Introduction

It is well-known that alkylated and N-alkylated pyrroles were obtained from condensation between 2,5-hexanedione and amines (*1*). The UV (*1*, *2*), IR, and NMR spectra (*1*, *3*) and mass fragmentation (*1*, *4*, *5*) of pyrrole and some of its derivatives have been investigated previously. Our N-substituted pyrroles series allows us to establish a correlation study between the UV and NMR, a study not previously attempted on pyrroles. The mass spectra of these derivatives have also been measured.

Results and Discussion

The position of CH_3 groups in the NMR spectra (Table I) of Ib ($\delta = 2.008$) and Id ($\delta = 1.930$) are shifted to a higher field compared with that of Ia ($\delta = 2.090$) owing to the conjugation of the nitrogen electron pair with the π-conjugated system of the phenyl ring. *N*-Aryl substitution causes a red shift of the long UV band (Table I) by about the same amount, 28 ± 3 nm, which probably means that only 28/240 = 0.117 or 11.7% of the total electronic density of the nitrogen electrons is conjugated with the aryl ring.

The long UV bands of all compounds are red-shifted in concentrated sulfuric acid by the same amount (Table I); in this medium withdrawal of the nitrogen electron lone pair should occur and the spectrum may represent a charged aminodiene (*6*).

Table I. ^{1}H NMR Data and UV Absorption Bands of 2,5-Dimethyl-N-substituted Pyrroles in Ethanol and Concentrated Sulfuric Acid

	NMR data			UV bands [λ_{max}, nm (log ϵ, m^2 mol^{-1})]	
molecule	δ, ppm	intens	multipl	EtOH	concd H_2SO_4
pyrrole[a]				240 (2.48)	
				210 (3.70)	
Ia	0.84	3	triplet	226 (2.10)[b]	242 (2.46)
	1.61	2	sextet	208 (2.30)	202 (2.21)
	2.09	6	singlet		
	3.55	2	triplet		
	5.62	2	singlet		
Ib	2.008	6	singlet	264 (2.31)	284 (2.73)
	5.88	2	singlet	230 (3.05)[b]	240 (2.86)
	7.20–7.33	5	complex	213 (2.23)	205 (3.12)
Ic	2.010	6	singlet	271 (2.04)[b]	294 (2.51)
	3.85	3	singlet	234 (3.03)	235 (2.64)[b]
	5.87	2	singlet	209 (3.02)	222 (2.70)
	6.91–7.23	4	complex		
Id	1.93	6	singlet	266 (2.40)	288 (2.72)
	5.79	2	singlet	243 (3.03)	242 (2.83)
	6.88–6.98	2	doublet	218 (3.28)	219 (3.07)
	7.23–7.32	2	doublet		
Ie				270 (3.23)	291 (3.35)
				204 (3.16)	208 (2.90)

[a] From ref 2. [b] Shoulder.

The mass spectra of compounds Ia–e are shown in Table II in the form of relative intensities vs. *m*/*z*.

Experimental Section

Compound Ia (Figure 1) was prepared by refluxing, for 90 min equimolar amounts of *n*-propylamine and 2,5-hexanedione;

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