NO2C6H4NHNH2, 100-16-3; HONH2+HCI, 5470-11-1; H2NC(O)NH2, 57-13-6; H_NC(S)NH_2, 62-56-6; 2,4-dinitrophenylhydrazine, 119-26-6.

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Synthesis and Properties of Some N,N-Dialkylamides as New Extractants

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Forty-seven new N,N-dialkylamides were prepared and their physical and spectral data are tabulated. The solubility of amides 1-47 in organic and aqueous solvents was tested and the results showed that as the amides are freely soluble in organic solvents they are nearly insoluble in aqueous medium. The results of the thermal stability of amides 1-47 at 80 °C, the chemical stability toward 3 M nitric acid, and the radiolytic stability toward γ -rays were found to be encouraging. The solubility and stability results obtained are useful for the preliminary evaluation of amides as new extracting agents.

Recently a systematic investigation of a number of amides and their application in nuclear processes was reported (1, 2). Some of these amides were proposed as alternative extractants to tributyl phosphate (TBP) for separation of some actinides from lanthanides (2). Moreover, it was reported that the extraction properties of certain dialkylamides toward some tetra- and hexavalent ions were greatly influenced by the structure of amide (1, 3). The present work covers the preparation and preliminary evaluation of different series of N,Ndialkylamides (1-47) as new extracting agents (Table I).

In general, the amides were obtained as oily compounds with good yields (62-98%), except for N,N-dicyclohexyl derivaties, where the yields ranged from 35 to 40% and the compounds were solids. The physical and spectral data are tabulated (Table II).

Solubility Behaviors. With the exception of the N,N-dicyclohexyl derivatives, the extent of the solubility of the amides in n-dodecance, mesitylene, chloroform, and ethanol was found to be more than 3 mol/L. On the other hand, the solubility in water was generally less than 10⁻³ g/L (Table III). From the solubility behavior point of view, the above results are considered to be encouraging.

Thermal, Chemical, and Radiolytic Stabilities. The results indicated in Table III show that the amides 1-47 are considered to be thermally, chemically, and radiolytically stable enough to be proposed as extracting agents for metal ions. In general, the above solubility and stability results are parallel to those reported for TBP as well as for some other amides which were recommended recently as good extractants for some actinide elements (1, 2). In fact, these results fulfill some of the required features for good extractants (4). The results also re-

Table I. Preparation and Notation of N,N-Dialkylamides

 $RCOCl + HNR'_2 \xrightarrow{Et_3N}$ RCONR + Ft. NUCI

	RCONK ₂ + Et ₃ NHC	,1 	
amide	R	R′	
1	C _e H ₁₁ (CH ₂) ₂	n-Pr	
2	CaH11(CH2)	n-Bu	
3	CaH11(CH2)	sec-Bu	
4	C _a H ₁₁ (CH ₀)	C.H.	
5	CeH ₁₁ (CH ₀)	n-Pr	
6	C _e H ₁₁ (CH ₀) ₀	i-Pr	
7	C _e H ₁₁ (CH _e) ₂	n-Bu	
8	CeH ₁₁ (CH ₂)	i-Bu	
ğ	C _a H ₁₁ (CH _a) _a	sec-Bu	
10	C _a H ₁₁ (CH _a) _a	n-Her	
11	CoHu(CHo)	C.H.	
12	C.H.CH.	n-Pr	
13	C.H.CH.	n-Bu	
14	C.H.CH.	80C-B11	
15	C.H.CH.	C.H.	
16	CuHa	Et	
17	C_1H_2	n-Pr	
18	C_{11}	n-Bu	
19		sec-Bu	
20	CueHan	n-Bu	
21	CueHen	sec.Bu	
22	CH ₂ (CH ₂)	n-Bu	
23	CH ₂ (CH ₂)	i-Bu	
20	CH ₂ (CH ₂)	sec-Bu	
25	CH ₂ (CH ₂)	n-Her	
26	(CH _a) _a CCH _a	n-Bu	
27	(CHa) CCH	sec-Bu	
28	$(CH_{a})_{a}CCH_{a}$	n-Hex	
29	Ph(CH _a)a	n-Pr	
30	$Ph(CH_{a})_{a}$	n-Bu	
31	$Ph(CH_{a})_{a}$	sec-Bu	
32	$Ph(CH_a)_a$	C.H.	
33		n-Pr	
34	Ph(CH _a) _a	n-Bu	
35	Ph(CH _a)	sec-Bu	
36	$Ph(CH_{a})_{a}$	C.H.	
37	Ph(CH _a)	<i>n</i> -Pr	
38	Ph(CH _a)	n-Bu	
39	Ph(CHa)	sec-Bu	
40	Ph(CHa)	CeH11	
41	EtCHPh	n-Pr	
42	EtCHPh	n-Bu	
43	EtCHPh	sec-Bu	
44	EtCHPh	C_6H_{11}	
45	$MeCHPhCH_2$	n-Pr	
46	MeCHPhCH ₂	n-Bu	
47	MeCHPhCH	sec-Bu	

Table II.	Physical	and Spectral	Data	of N,N	V-Dialkylamides ^a
	hn °C/		0 b	vield	$IR(\mu_{\alpha}, \alpha)^{c}$

amide	mmHg	$n_{\rm D}$ (T, °C)	ρ, \tilde{c}	yiela, %	cm^{-1}	NMR δ^d
1	152/07	1 4746 (21)	0.810	61.7	1634	32 († 4 H CH-NCH.) 2 18 († 2 H COCH.) 1 98-0 87
2	138/01	1 4730 (20)	0.826	72.4	1636	(m, 23 H, rest of protons) 32 (t. 4 H, CH_NCH_) 2.2 (t. 2 H, COCH_, 0.92 (d. 6 H, 2 CH_))
-	142/0.3	1.4764 (20)	0.826	77.6	1632	1.95–1.17 (m, 11 H, rest of protons) 3.63 (m, 1 H, NCH), 2.94 (m, 1 H, NCH), 2.2 (t, 2 H, COCH ₂),
4	thist ail	1 4020 (95)		95	1696	2.02-0.34 (m, 29 H, rest of protons)
4 5	145/0.1	1.4930 (28)	0.833	35 86.0	1635	3.3 - 0.7 (m, an protons) 3.2 (t, 4 H, CH ₂ NCH ₂), 2.16 (t, 2 H, COCH ₂), 1.98-0.88 (m, 25 H, rest of protons)
6	124/0.01	1.4613 (24)	0.832	81.1	1640	3.7 (m, 2 H, CHNCH), 3.0–0.6 (m, 29 H, rest of protons)
7	143/0.04	1.4719 (23)	0.831	89.6	1640	3.2 (t, 4 H, CH ₂ NCH ₂), 2.13 (t, 2 H, COCH ₂), 1.95–0.34
8	137/0.01	1.4600 (24)	0.863	90.0	1642	(m, 29 H, rest of protons) 3.13 (d, 4 H, CH ₂ NCH ₂), 2.18 (t, 2 H, COCH ₂), 2.0–0.5
9	147/0.15	1.4743 (23)	0.826	83.0	1639	(m, 29 H, rest of prowns) 3.6 (m, 1 H, NCH), 2.94 (m, 1 H, NCH), 2.18 (t, 2 H, COCH ₂), 1 98-0 35 (m, 31 H, rest of protons)
10	157/0.01	1.4603 (24)	0.840	92.0	1638	3.18 (t, 4 H, CH_2NCH_2), 2.15 (t, 2 H, $COCH_2$), 1.28 (m, 37 H, rest of protons)
11	mp 52–54 (<i>n</i> -hexane)			40.0	1630	3.9-0.5 (m, all protons)
12	101/0.07	1.4690 (22)	0.828	80.6	1631	3.2 (t, 4 H, CH ₂ NCH ₂), 2.23 (d, 2 H, COCH ₂), 0.88 (t, 6 H, 2 CH ₂), 2.08-1.15 (m, 13 H, rest of protons)
13	116/0.08	1.4680 (22)	0.817	81.8	1630	3.23 (t, 4 H, CH ₂ NCH ₂), 2.21 (d, 2 H, COCH ₂), 0.94 (d, 6 H, 2 CH ₃), 2.06-1.12 (m, 17 H, rest of protons)
14	116/0.2	1.4711 (22)	0.797	66.3	1628	3.65 (m, 1 H, NCH), 2.93 (m, 1 H, NCH), 2.23 (d, 2 H, COCH ₂), 1.23 (m, 25 H, rest of protons)
15	mp 65-67 (<i>n</i> -hexane)	1 (005 (00)	0.995	40.0	1632	3.8-0.7 (m, all protons)
16	130/0.1	1.4885 (26)	0.835	90.13	1630	3.3 (t, 4 H, CH_2NCH_3 , 2.57 (m, 1 H, COCH), 1.49 (s, 20 H, cyclic protons); 1.10 (t, 6 H, 2 CH ₃)
17	140/0.08	1.4855 (26)	0.829	92.97	1629	3.20 (t, 4 H, CH ₂ NCH ₂), 2.60 (m, 1 H, COCH), 1.46 (s, 20 H, cyclic protons); 4 H, 2 CH ₂), 0.90 (t, 6 H, 2 CH ₃)
18	150/0.15	1.4844 (24)	0.917	92.48	1630	3.20 (t, 4 H, CH ₂ NCH ₂), 2.60 (m, 1 H, COCH), 1.46 (s, 20 H, cyclic protons); 8 H, 4 CH ₂), 0.96 (t, 6 H, 2 CH ₃)
19 20	140/0.1 155/0.09	1.4888 (26) 1.4729 (23)	$0.803 \\ 0.829$	83.37 94.1	1630 1635	2.60 (m, 2 H, CHNCH), 3.3–0.7 (m, 37 H, rest of protons) 3.23 (t, 4 H, CH ₂ NCH ₂), 2.60 (m, 1 H, COCH), 1.40 (s, 22 H,
21	145/0.04	waxy		73.13	1630	cyclic protons); 8 n, 4 CH_2), 0.95 (t, 6 n, 2 CH_3) 2.8 (m, 1 H, NCH), 2.74 (m, 1 H, NCH; 1 H, COCH) 2–0.7 (m, 38 H, rest of protons)
22	112/0.1	1.4393 (25)	0.810	86.76	1640	(III, 50 4 H, CH ₂ NCH ₂), 2.18 (t, 2 H, COCH ₂), 1.32 (s, 16 H, 8 CH ₂), 0.93 (d, 9 H, 2 CH ₃)
23	126/0.5	1.4381 (25)	0.811	78.99	1649	3.3-2.87 (d, 4 H, CH ₂ NCH ₂), 2.24 (t, 2 H, COCH ₂), 0.94 (d, 15 H, 5 CH ₃), 2.0-1.1 (m, 8 H, 4 CH ₂ ; 2 H, 2 CH)
24	124/0.3	1.4426 (23)		97.60	1640	3.64 (m, 1 H, NCH), 2.96 (m, 1 H, NCH), 2.18 (t, 2 H, COCH ₂), 1.30 (d, 12 H, 6 CH ₂ ; 6 H, 2 CH ₃), 0.88 (d, 9 H, 3 CH ₃)
25 26	140/0.05 120/0.2	1.4431 (25) 1.4339 (28)	0.819 0.833	87.60 80.87	$\begin{array}{c} 1640 \\ 1632 \end{array}$	3.20 (t, 4 H, CH_2NCH_2), 2.18 (t, 2 H, $COCH_2$), 1.30 (s, 24 H, 12 CH_2) 3.23 (t, 4 H, CH_2NCH_2), 2.10 (s, 2 H, $COCH_2$), 1.43 (m, 8 H, 4 CH_2), 1.00 (c, dH, (CH, Q), 0.00 (c, dH, CCH_2), 1.43 (m, 8 H, 4 CH_2),
27	96/0.15	1.4371 (26)	0.805	66.37	1632	1.03 (s, 9 H, $(CH_3)_{3}$ C), 0.93 (s, 6 H, 2 CH ₃) 3.64 (m, 1 H, NCH), 3.0 (m, 1 H, NCH), 2.10 (s, 2 H, COCH ₂), 18-07 (m, 25 H, rest of protons)
28	135/0.25	1.4381 (27)	0.852	83.88	1638	3.20 (t, 4 H, CH_2NCH_2), 2.07 (s, 2 H, $COCH_2$), 1.32 (s, 16 H, 8 CH_2), 1.02 (s, 9 H, $(CH_2)CC_1$), 0.90 (s, 6 H, 2 CH_2)
29	126/0.15	1.5080 (24)	0.905	87.00	1640	7.1 (s, 5 H, ArH), 3.4-2.27 (m, 4 H, CH ₂ NCH ₂ ; 4 H, COCH ₂ CH ₂)
30	135/0.08	1.5150 (25)	0.870	80.00	1635	7.1 (s, 5 H, ArH), $3.43-2.23$ (m, 4 H, CH ₂ NCH ₂ ; 4 H, COCH ₂ CH ₂),
31	128/0.08	1.5570 (25)	0.915	75.00	1630	1.5–1.1 (m, 5 H, 4 CH_2), 0.50 (d, 6 H, 2 CH_3) 7.1 (s, 5 H, ArH), 3.55–2.23 (m, 2 H, CHNCH; 4 H, COCH ₂ CH ₂), 0.83 (t, 6 H, 2 CH_3), 1.97–1.05 (m, 10 H, rest of protons)
32	mp 82-84 (<i>n</i> -hexane)			66.0	1635	7.1 (s, 5 H, ArH), 3.0-0.6 (m, 26 H, rest of protons)
33	129/0.2	1.4930 (25)	0.900	76.0	1625	7.1 (s, 5 H, ArH), 3.1 (q, 4 H, CH ₂ NCH ₂), 2.6 (t, 2 H, COCH ₂), 0.83 (t, 6 H, 2 CH ₃), 2.35-1.08 (m, 8 H, rest of protons)
34	136/0.1	1.4990 (27)	0.910	66.0	1620	7.1 (s, 5 H, ArH), 3.13 (q, 4 H, CH ₂ NCH ₂), 2.6 (t, 2 H, COCH ₂), 2.4–0.9 (m, 18 H, rest of protons)
35	136/0.15	1.5030 (25)	0.910	70.8	1640	7.1 (s, 5 H, ArH), 3.4 (m, 2 H, CHNCH), 2.7 (t, 2 H, COCH ₂), 2.4-1.0 (m, 20 H, rest of protons)
36	183/0.15	1.5280 (31)	0.940	71.5	1640	7.1 (s, 5 H, ArH), 3.2 (m, 2 H, CHNCH), 2.66 (t, 2 H, COCH ₂), 2.34–0.6 (m, 24 H, rest of protons)
37	145/0.08	1.5030 (27)	0.810	86.1	1640	7.1 (s, 5 H, ArH), 3.13 (t, 4 H, CH_2NCH_2), 2.6 (t, 2 H, $COCH_2$), 2.17 (t, 2 H, $PhCH_2$, 1.60 (m, 8 H; 4 CH_2), 0.83 (t, 6 H, 2 CH_3)
38	158/0.1	1.5020 (21)	0.780	85.0	1630	7.1 (s, 5 H, ArH), 3.16 (m, 4 H, CH_2NCH_2), 2.6 (t, 2 H, $COCH_2$), 2.16 (t, 2 H, $PhCH_2$), 1.93–1.1 (m, 12 H, 6 CH_2), 0.93 (d, 6 H, 2 CH_3)
39	150/0.07	1.5019 (27)	0.890	65.3	1640	7.1 (s, 5 H, ArH), 3.5 (m, 1 H, NCH), 2.92 (m, 1 H, NCH), 2.58 (t, 2 H, COCH ₂ , 2.17 (t, 2 H, PhCH ₂), 1.9–0.6 (m, 20 H, rest of protons)
40	184/0.06	1.5028 (26)	0.760	73.5	1643	7.1 (s, 5 H, ArH), 3.28 (m, 2 H, CHNCH), 2.6 (t, 2 H, COCH ₂), 2.17 (t, 2 H, PhCH ₂), 1.6 (m, 24 H, rest of protons)

Table II (Continued)

amide	bp, °C/ mmHg	$n_{\rm D}~(T, ^{\rm o}{\rm C})$	$ ho,^b$ g/mL	yield, %	$\operatorname{IR}_{\operatorname{cm}^{-1}}^{(\nu_{\mathrm{C}} - 0),^{c}}$	NMR δ^d
41	112/0.08	1.5079 (25)	0.810	61.5	1640	7.2 (s, 5 H, ArH), 3.2 (m, 4 H, CH ₂ NCH ₂ ; 1 H, COCH), 2.54–0.38 (m, 15 H, rest of protons)
42	129/0.1	1.4961 (29)	0.840	86.4	1620	7.16 (s, 5 H, ArH), 3.16 (m, 4 H, CH ₂ NCH ₂ ; 1 H, COCH), 2.46-0.46 (m, 19 H, rest of protons)
43	118/0.08	1.5062 (29)	0.840	63.5	1640	7.16 (s, 5 H, ArH), 4.13-3.17 (m, 2 H, CHNCH), 2.77 (m, 1 H, COCH), 2.46-0.13 (m, 21 H, rest of protons)
44	mp 90-91 (<i>n</i> -hexane)			88.0	1615	7.23 (s, 5 H, ArH), 3.47 (t, 2 Ĥ, CHNCH), 2.7 (m, 1 H, COCH)
45	153/0.2	1.4933 (26)	0.810	90.05	1635	7.14 (s, 5 H, ArH), 3.7-2.7 (m, 4 H, CH ₂ NCH ₂ ; 1 H, PhCH), 2.44 (d, 2 H, COCH ₂), 1.43 (m, 4 H, 2 CH ₂ ; 3 H, PhCCH ₃), 0.8 (t, 6 H, 2 CH ₃)
46	148/0.16	1.4873 (26)	0.840	91.10	1633	7.13 (s, 5 H, ArH), 3.5–2.7 (m, 4 H, CH ₂ NCH ₂ ; 1 H, PhCH), 2.4 (d, 2 H, COCH ₂), 1.4 (d, 8 H, 4 CH ₃ ; 3 H, PhCCH ₃), 0.88 (d, 6 H, 2 CH ₃)
47	134/0.12	1.4920 (26)	0.910	94.24	1625	7.16 (s, 5 H, ArH), 4.0–2.67 (m, 2 H, CHNCH; 1 H, PhCH), 2.45 (d, 2 H, COCH ₂), 1.24–1.6 (m, 3 H, PhCCH ₃), 1.33 (d, 4 H, 2 CH ₂ ; 6 H, 2 CH ₃), 0.87 (t, 6 H, 2 CH ₃)

^aElemental analyses for C, H, and N in agreement with theoretical values were obtained and submitted for review. ^bDensity. ^cLiquid film. ^dIn CCl₄.

Table III. Properties o	of N .	N-Dialk	vlamides	(1-47)
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property	result
solubility in organic solvents solubility in aqueous phase	>3 mol/L <10 ⁻³ g/L
density in mesitylene	0.76-0.94 g/mL No sign of degradation was
thermal stability	observed when the amide was left at 80 °C for 2 days.
chemical stability	No sign of degradation was observed after contacting equal volumes of 1 M amide in mesitylene with 3 M HNO, for 1 month
radiolytic stability	In most cases less than 5% and 10% degradations were observed at 15 and 30 Mrd, respectively.

vealed that the solubility and stability factors are independent of the structure of amides.

The main advantages of this class of extractants are their easy preparation as well as the fact that their degradation products, if any, are easily washed and do not interfere during the extraction process of metal ions (3). In fact, one of the main disadvantages of N,N-dlalkylamides is their tendency to form a third phase during the extraction process of uranium from nitric acid medium. This behavior is highly dependent on the structure of the dialkylamides (1, 3) and it is, in fact, our curent scope of interest.

Experimental Section

Infrared spectra were recorded on an IR 10 Beckman infrared spectrophotometer and ¹H NMR spectra with a Varian T 60 A spectrometer using Me_4Si as internal standard. C, H, N elementary analyses were obtained by using a 185 HP anatyzer.

Preparation of N, N-Dialkylamides. The amides 1-47 were prepared and purified by redistillation under vacuum following the general reported procedure (5). The physical and spectral data are tabulated (Table II).

Determination of Amides. The concentrations of *N*,*N*-dialkylamides were determined by potentiometric titration with perchloric acid in acetic anhydride (β). A metrohm E 526 potentiography connected to a dosimat piston buret E 535 with a swingout magnetic stirrer E 549 was used. A metal electrode EA.2A filled with LiCIO₄ (0.1 M) as a salt bridge was used for the end-point titration.

Solubility Measurements. The extent of amide solubility in organic solvents was carried out by dissolving an excess of each amide in the appropriate solvents (*n*-dodecane, mesitylene, chloroform, ethanol); the concentrations of the solutions were determined by potentiometric measurements (Table III). The solubilities of the amides in aqueous phase were checked by GLC technique. The densities of the amides were also determined and tabulated (Table II).

Thermal Stability. The thermal stabilities of the amides (1-47) was carried out by leaving a known weight of each amide in a thermostated bath at 80 °C. The concentrations of the amides were followed as a function of time over a period of 2 days (Table III).

Chemical Stability. The chemical stabilities of the amides (1-47) toward nitric acid were carried out by contacting their mesitylene solutions (1 M) with nitric acid (3 M) in a thermostated shaking bath set at 25 °C. The concentration of each amide was followed as a function of time over a total period of 1 month (Table III).

Radiolytic Stability. The effect of different γ -dose (from ⁶⁰Co cell) on a solution of amide in mesitylene (1 M), which was equilibrated with nitric acid (3 M), was followed and the general results are indicated in Table III.

Registry No. 1, 72299-19-5; 2, 72299-20-8; 3, 91424-53-2; 4, 91424-54-3; 5, 72299-33-3; 6, 91424-55-4; 7, 72299-34-4; 8, 91424-56-5; 9, 91424-57-6; 10, 91424-58-7; 11, 91424-59-8; 12, 91424-60-1; 13, 91424-61-2; 14, 91424-62-3; 15, 91424-63-4; 16, 91424-64-5; 17, 91424-65-6; 18, 91424-66-7; 19, 91424-67-8; 20, 91424-68-9; 21, 91424-60-0; 22, 57303-22-7; 23, 57303-37-4; 24, 53463-20-0; 25, 91424-70-3; 26, 29846-86-4; 27, 91424-71-4; 28, 91424-72-5; 29, 91424-76-6; 30, 57772-72-2; 31, 91424-71-4; 28, 91424-78-8; 31, 91424-76-9; 34, 61123-41-9; 35, 91424-77-0; 36, 91424-78-1; 37, 91424-79-2; 38, 91424-80-5; 39, 91424-81-6; 40, 91424-82-7; 41, 91424-83-8; 42, 91424-87-2; 47, 91424-88-3.

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