and osmotic coefficients in dilute solutions of some 2–1 electrolytes, including chlorides, bromides, and perchlorates, are calculated and compiled in Tables II, III, and IV. The maximum estimated error for these values is about 0.3%.

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NEW COMPOUND SECTION

The Synthesis of Derivatives of 3,7-Diazanonanedioic Acid

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The synthesis of several esters of 2,8-disubstituted 3,7-diazanonanedioic acid from amino acid esters and 1,3-dibromopropane is described. The compounds reported include ethyl 3,7-diaza-2,8-di(isobutyl)nonanedioate, methyl 3,7-diaza-2,8-(3-thiabutyl)nonanedioate, ethyl 3,7-diaza-2,8-dimethylnonanedioate, ethyl 3,7-diaza-2,8-dibenzylnonanedioate, and benzyl 3,7-diaza-2,8-dimethylnonanedioate.

As part of a study of multidentate ligands, it was desired to prepare symmetrical amino esters of the general formula $(XO_2CCHRNHCH_2)_2CH_2$ (I), where R could be the side chain groups of the naturally occurring amino acids. We wish to report the synthesis of several of these esters of 2,8-disubstituted 3,7-diazanonanedioic acid by a simple, direct route (*1*).

A mixture of two parts amino acid ester hydrochloride, one part 1,3-dibromopropane, and four parts triethylamine was heated for several days in a suitable solvent. The product was isolated by extraction and purified by chromatography. The structures of the resulting di(amino esters) (Table I) were confirmed by spectral data and analysis of the dihydrochloride derivatives.

$$BrCH_2CH_2CH_2Br + 2NH_2CHRCO_2X \xrightarrow{Et_3N} XO_2CCHRNHCH_2CH_2CH_2NHCHRCO_2X$$

When exposed to the atmosphere, these compounds are stable for several days. After this time a precipitate slowly forms in the oils. Infrared analysis of this precipitate shows amine salt and carboxylate peaks, indicating that it is a hydrolysis product.

Table I. Derivatives of 3,7-Diazanonanedioic Acid $XO_2CCHRNHCH_2CH_2CH_2NHCHRCO_2X$

Compound	R	X	Solvent	Yield (%)	Mp of dihydrochloride (°C)
1a	H–	Et–	EtOH	23	——[a]
b	$(CH_3)_2CHCH_2$–	Et–	EtOH	51	178–180
c	$CH_3SCH_2CH_2$–	Me–	MeOH	35	158–160
d	CH_3–	Et–	EtOH	56	151–153
e	$C_6H_5CH_2$–	Et–	EtOH	50	168–169
f	CH_3–	$C_6H_5CH_2$–	Dioxane	29	243–245

[a] An analytically pure sample of this compound could not be obtained.

Table II. IR Data for Compounds Ia–f

Compound	IR data (thin film), cm^{-1}			
	N—H	C=O	C—O	Other absorptions
Ia	3400	1748	1170	950, 910, 755
b	3380	1730	1170	935, 860, 755
c	3390	1736	1050	990, 915, 900, 760-750
d	3356	1727	1170	1030, 860, 755
e	3352	1730	1170	1090, 755, 700
f	3390	1730	1170	740, 695

Table III. NMR Data for Compounds Ia–f[a] $(XO_2CC(R)H^1NHCH^2_2)_2CH^3_2$

Compound	H^1	H^2	H^3	X	R
Ia	3.37 s	2.70 dt	1.65 m	1.28 t (CH_3–) 4.19 q (–CH_2O–)	——[b]
b	2.98 t	2.34 dt	1.5 m	1.09 t (CH_3–) 3.96 q (–CH_2O–)	0.80 d (–$CH(CH_3)_2$) 1.0–1.5 bm (–CH_2CH–)
c	3.29 t	2.54 m	1.5 m	3.64 s	1.9 m (–CH_2–) 2.02 s (–SCH_3) 2.5 m (–CH_2S–)
d	3.29 t	2.50 dt	1.52 m	1.16 t (CH_3–) 4.07 q (–CH_2O–)	1.16 d (CH_3–)
e	3.20 t	2.34 t	1.34 m	0.93 t (CH_3–) 3.82 q (–CH_2O–)	2.67 d (–$CH_2C_6H_5$) 7.03 s (C_6H_5–)
f	3.20 t	2.41 dt	1.35 m	4.91 s (–$OCH_2C_6H_5$) 7.11 s (C_6H_5–)	1.10 d (CH_3–)

[a] Recorded in $CHCl_3$ or CCl_4. Reported in ppm (δ) downfield from Me_4Si: d = distorted, b = broad. [b] A small impurity peak is present at 3.48.

Experimental Section

Melting points were determined on a Fisher-Johns apparatus and are uncorrected. NMR spectra were determined on a Hitachi-Perkin Elmer R-24 and a Varian T-60 NMR spectrophotometer. IR spectra were determined on a Perkin-Elmer Model 137 spectrophotometer. Analyses were performed by Instranal Laboratories and were submitted for review.

Typical Procedure. The hydrochloride of the ester of an amino acid (0.05 mol) was added to a solution of 0.025 mol of 1,3-dibromopropane and 0.10 mol of triethylamine in ethanol. This mixture was refluxed for 5 days, during which time it slowly turned orange. The reaction mixture was concentrated, added to water, adjusted to pH 12 with NaOH, and extracted with ether. Evaporation of the ether gave an oil, which was purified by chromatography on silicic acid (3)/celite (1). The purified product was a straw-colored oil. The dihydrochloride derivatives were prepared by dissolving the product in ethanol and adding a solution of ethanol saturated with HCl gas. The dihydrochloride was precipitated by the addition of ether and recrystallized from ethanol-ether mixtures.

Infrared data for these compounds are presented in Table II. The NMR data are shown in Table III.

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(1) Adapted from Buehler, C. A., Pearson, O. E., "Survey of Organic Synthesis," Wiley, New York, N.Y., 1970, pp 444–446.

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Correction

For the paper, "Preparation of New 2-Pyridyl and Pyrazinylhydrazones Containing Ferroin Group", by Francis H. Case (*J. Chem. Eng. Data*, **21**, 124 (1976)), on page 124, Number XIII under the structural formula should be XXI. XIII cited in the text is correct (line 10 from bottom of page). Also 2,2′-pyridyl (lines 6 and 10 from bottom) should be 2,2′-pyridil.