Notes

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Kōno Kinoshita and Shoichi Nakajima: Studies on the Structure of Itaconitin. III.[1] Identification of Organic Acids as *p*-Aminoazobenzene Derivatives. (2).

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With a view to determining the acids, obtained as the decomposition product from itaconitin derivatives, a new method for identification of fatty acids was devised as reported in the preceding paper.[1] Attempt was made for the application of this method to other ordinary organic acids.

The reported procedure for preparing 4-aminoazobenzene derivatives was to heat the acids with 4-aminoazobenzene at various temperatures but this procedure was found not to be applicable to any kind of acid derivatives. The acids were therefore derived to acyl chlorides and reacted with 4-aminoazobenzene to form the objective derivatives. All the 4-aminoazobenzene derivatives prepared had a high melting point and orange color.

Table I.

4-Aminoazobenzene N-acyl deriv.	m.p. (°C)	Appearance (Orange color)	Solvent of recrystn.	Mol. formula	Analysis (%) Calcd. C	Calcd. H	Calcd. N	Found C	Found H	Found N
Isovaleryl	179	needles	benzene–petr. benzine	$C_{17}H_{19}ON_3$	72.57	6.81	14.94	72.02	6.62	14.90
Monochloroacetyl	155	plates	benzene	$C_{14}H_{12}ON_3Cl$	61.43	4.42	15.32	61.66	4.45	15.53
Dichloroacetyl	177	prisms	petr. benzine	$C_{14}H_{11}ON_3Cl_2$	54.58	3.60	13.65	54.34	3.49	13.64
Lauroyl	124	plates	Me_2CO	$C_{24}H_{33}ON_3$	75.95	8.76	11.07	75.91	8.40	11.29
Benzoyl	205	〃	〃	$C_{19}H_{10}ON_3$	75.73	5.12	13.73	76.11	5.02	13.95
Cinnamoyl	191	〃	EtOH	$C_{21}H_{17}ON_3$	77.04	4.90	12.54	76.95	5.23	12.84
p-Nitrobenzoyl	275	needles	Me_2CO	$C_{19}H_9O_3N_4$	65.89	4.07	16.18	65.80	3.94	16.24
Benziloyl	195	rhombic crystals	petr. benzine	$C_{26}H_{21}O_2N_3$	76.64	5.20	10.31	77.04	5.36	10.26
Diphenylacetyl	200	needles	〃	$C_{26}H_{21}ON_3$	79.77	5.41	10.74	79.37	5.59	10.69
Nicotinoyl	205	plates	EtOH	$C_{18}H_{14}ON_4$	71.51	4.67	18.53	71.27	4.65	18.66
Isonicotinoyl	239	〃	〃	$C_{18}H_{14}ON_4$	71.51	4.67	18.53	70.76	4.80	18.61

Experimental[*2]

Preparation of 4-Aminoazobenzene Derivatives of Organic Acids—Each acid was dissolved in 5 parts of $SOCl_2$ and boiled for 0.5 hr. After the reaction, excess $SOCl_2$ was removed in vacuum and Me_2CO solution of 4-aminoazobenzene was added to the reaction product. After boiling this mixture for 30 min., the solvent was evaporated to leave a crystalline brei of crude 4-aminoazobenzene derivative, which was recrystallized from the solvent specified in Table I. In some cases alumina column was used for purification of the product.

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Summary

Some 4-acylaminoazobenzenes, the colored derivatives of organic acids, were made further for the purpose of the identification of the acids. The preparation procedure was somewhat modified.

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*2 All melting points are uncorrected.

1) Part II: This Bulletin, 8, 56(1960).