

N-BENZYLAMIDES AS DERIVATIVES FOR  
IDENTIFICATION OF THE ACYL GROUP IN  
ESTERS

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IDENTIFICATION OF THE ACYL GROUP IN  
ESTERS

By

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## INTRODUCTION

A method for characterizing the acyl group in esters which would involve but a single reaction, use readily available reagents, and prove generally applicable could be regarded as a decided improvement in the identification of esters.

This investigation constitutes an attempt to improve and extend such a method first suggested by the work of Buehler and Mackenzie (1).

## HISTORICAL

The standard method of preparing derivatives for identification of the acyl group in esters consists of the following steps: saponification of the ester, isolation of the acid or its salt, and preparation of the desired derivative, for which many procedures are available (2). The method is rather lengthy and often proves unsatisfactory at one of these stages.

Two methods have been proposed for preparing a derivative directly from the ester. Koelsch and Tenenbaum (3) obtained p-toluides by adding the ester to a derivative of a Grignard reagent prepared from ethylmagnesium bromide and p-toluidine. Hardy (4) used aniline to obtain anilides by the same procedure.

Buehler and Mackenzie (1) employed direct aminolysis of esters in aqueous solution to obtain N-benzylamides using benzylamine as the aminolyzing agent.



They applied the method to twelve esters and found it to be unsatisfactory for those of the higher fatty acids. It likewise proved undesirable for esters of halogenated acids since the derivative usually obtained was the non-specific benzylammonium halide.

Buehler and Mackenzie are the only workers to study the aminolysis of esters as a means of identification, and indeed a survey of the literature reveals that comparatively little systematic investigation of the reaction has been done for any reason.

Mailhe and Bellegarde (5) report that substituted amides are

readily produced when esters and amines are allowed to react in the presence of  $\text{Al}_2\text{O}_3$  or  $\text{ThO}_2$  at  $480-900^\circ$ .<sup>2</sup>

Audrieth and co-workers (6,7) have found that the aminolysis of esters is catalyzed by alkylammonium salts, especially the chlorides and nitrates.

Turner and co-workers (8,9,10) have employed piperidine to aminolyse various substituted aryl p-toluenesulfonates. They were interested in regaining the substituted phenol, the hydroxyl group of which had been protected during nitration by esterification with p-toluenesulfonic acid.

Kurtz and Wilson (11) have suggested the use of saccharolactone as a reagent for the separation of amines as  $\text{N,N}'$ -substituted saccharamides.

Heubaum (12) makes the observation that aryl esters are more easily aminolysed than ethyl esters.

The reaction of aniline with ethyl malonate to give malonamide and ethyl malonanilate has been reported by Chattaway and Olmstead (13).

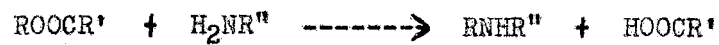
Murray and Dains (14) have prepared gamma-benzylallophanic ester by heating allophanic ester with benzylamine for three hours at  $135^\circ$ . Five hours heating at  $150^\circ$  gave sym-dibenzylbiuret.

Ryan and Casey (15) have found that when nitric esters are allowed to react with primary aromatic amines, "nitrates of the amines" are formed, but with secondary and tertiary amines, oxidation of the amine usually occurs.

Smirnov (16) states that isoamyl nitrite yields nitrosodibenzylamine when made to react with dibenzylamine. A similar reaction takes place with isoamyl nitrite and diphenylamine.



Irving (17) and Bell (18) have observed a less common splitting of certain esters by amines, so as to produce N-alkylation instead of N-acylation:



where R is substituted by highly negative groups such as halogen or the nitro group.

## EXPERIMENTAL

## Establishing the Method

The first part of this research was spent in trying to determine the proper reagents and conditions for the aminolysis of esters such that the method would be readily applicable to qualitative organic analysis. All experiments were carried out with small amounts of material to simulate the conditions one would have in an actual identification. All reactions were done in eight-inch pyrex test tubes in which were suspended finger condensers. These condensers were connected in series so that several could be run using a single water connection.

Choice of Amine.

The amine should boil at a relatively high temperature without decomposition, so that the reaction will proceed rapidly in the refluxing amine. It should be inexpensive, and for reasons which will be discussed later, water soluble. In addition, it is of course necessary that it yield solid derivatives with sharp melting points having sufficient scattering to permit easy differentiation.

Amines which seem to fit the above conditions best are: benzylamine, cyclohexylamine, and ethylenediamine. Trial runs with each of the above amines were made with ethyl acetate and ethyl benzoate using 1 ml. of the ester to 2 of the amine, and refluxing for one hour.

The mixtures were poured into water and shaken to remove the unreacted amine, then chilled in an ice bath. The substituted amides if formed in sufficient amounts would then crystallize. Satisfactory yields were obtained under the above conditions with benzylamine only. The products were proved to be the desired derivatives by recrystallization

from an acetone-water mixture and comparison of their melting points with literature values.

At one time dibenzylamine and dicyclohexylamine were considered for use, as both have rather high boiling points. However, the idea was discarded because their own slight solubility in water as well as that of their hydrochlorides makes their separation from the reaction mixture too difficult.

It is perhaps well before proceeding further to point out the analogies among such reactions as aminolysis, ammonolysis, and hydrolysis:



and also the applications of any of the above reactions to esters, amides, and acids:



Such reactions are termed solvolytic reactions by Audrieth (7) and he presents evidence to show similarities among them.

Considering the above, further evidence is to be found in the literature to support the choice of benzylamine for aminolysis of esters. Smith and Adkins (19) in their study of the relative reactivities of amines in the aminolysis of amides found benzylamine to rank high in this respect being surpassed only in small degree by several amines

which are more expensive and are less desirable for other reasons.

Benzylamine is miscible with water in all proportions, has a boiling point of 185°, and costs but 2 cents per gram when purchased in 500 gram lots.

#### Solvent.

Buehler and Mackenzie made their investigation with benzylamine in aqueous solution using equal parts of amine and water (ethyl alcohol or dioxan was added when necessary to effect complete miscibility). Runs were made in our work to determine the effectiveness of this procedure.

Mixtures containing 1 ml. of ethyl benzoate and 2 ml. of benzylamine with the addition of quantities of water varying from two drops to 10 ml. were refluxed for one hour. In no case did the yield of N-benzylbenzamide, obtained by pouring reaction mixtures into ice water and filtering, equal that given by the mixture of ester and amine without addition of water.

Benzylamine mixes readily with all esters tested so the addition of an inert organic solvent is unnecessary, and would only serve to dilute the reactants and thus increase the reaction time.

It was decided on the basis of the above facts to use no solvent at all in the aminolytic reactions.

#### Catalyst.

The catalytic effect of ammonium salts in the ammonolysis of esters has been observed by several investigators (20,21,22). Shatenshtein reports that the chloride is a better catalyst than the several other salts tried.

Ammonium salts have likewise been found to act as catalysts in the



aminolysis of esters (6,7). Audrieth and co-workers found the chloride and nitrate to give best results. It is generally thought that the substituted ammonium ion functions as the catalyst, yet the nature of the anion present may cause great variation in the catalytic properties of the salt, perhaps due to its degree of ionization in the amine or liquid ammonia. Audrieth noted that when ammonium chloride is added to n-butylamine or cyclohexylamine, ammonia is displaced from the salt by the amine, hence it is actually the amine hydrochloride which is functioning as catalyst.

Since Buehler and Mackenzie used no catalysts (unless water be considered so) in their work, it was believed that an improvement in their method might be made in this respect.

Runs of one hour length were made using mixtures of 1 ml. of ethyl benzoate and 2 ml. of benzylamine with one-tenth gram portions each of  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_4\text{Br}$ ,  $\text{NH}_4\text{I}$ ,  $\text{NH}_4\text{NO}_3$ , and  $(\text{NH}_4)_2\text{SO}_4$ , and also a mixture containing no catalyst. Increased yields of the amide were observed in all cases where a salt was present. Ammonium sulfate gave only a slight increase, very probably owing to its low solubility in boiling benzylamine. The chloride and nitrate were found to give the greatest increases, the yield being approximately doubled in each case.

Increasing the amount of catalyst above that specified in the preceding paragraph had no significant effect on the yield obtained.

## Testing the Method

### The Aminolysis.

When it was believed that a satisfactory method had been established, it remained to test it upon a variety of esters. The procedure used was as follows: 1 ml. of the liquid ester, or 1 gram if a solid, is added to 3 ml. of benzylamine together with 0.1 gram of ammonium chloride. The mixture is then refluxed for one hour. The evolution of ammonia is noticed as soon as the reactants become warm. Many of the esters require much less than an hour for completion of the reaction and form solids even at the refluxing temperature. It has been found, however, that one hour is sufficient time for practically all esters ordinarily encountered.

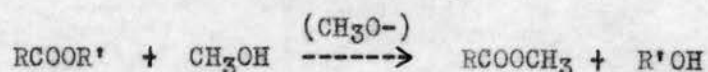
### Separation and Purification.

After reaction is completed the mixture is poured into 10 ml. of water, shaken to remove most of the unreacted amine, and chilled. In case no solid forms at this point, the addition of enough conc. HCl to completely neutralize all of the remaining amine will usually result in the amide's separating as a solid. The product is then isolated by filtration and washed with water. Washing with ligroin may then be done to remove any unreacted ester. None of the amides prepared were more than slightly soluble in ligroin whereas most of the esters were.

No single solvent was found suitable for recrystallization of all the amides. However, one of the following proved suitable: acetone, isopropyl alcohol (or either of these mixed with water), toluene, nitrobenzene, or occasionally water alone.

### Difficult Cases.

During the course of this investigation esters of the longer-chain aliphatic alcohols were found to be more difficult to aminolyze than the methyl or ethyl esters. Taylor and Baker (23) make the statement that methyl esters are much more reactive in ammonolysis than any others. Sessions (24) has found that conversion of longer-chain alkyl esters to methyl esters by reaction with methanol, termed methanolysis, greatly enhances the reactivity of these esters in subsequent conversion to amides by ammonolysis.



Fehlandt and Adkins (25) in their study of the replacement series of alkyl groups in alcoholysis have observed that methanol exceeds all other alcohols in its ability to cause substitution of methyl groups for other alkyl groups in esters. King (26) in investigating the rate of alcoholysis of aralkyl *p*-nitrobenzoates found that methanolysis proceeds much faster than ethanolysis.

All the above facts point to a way of dealing with esters of longer-chain alcohols. As a trial, 1 ml. each of amyl valerate and butyl benzoate, esters which had not given a solid derivative by the ordinary treatment, were heated separately for 30 minutes in 5 ml. of boiling methanol with a very small piece of sodium previously added (to form methoxide ion, which acts as a catalyst). The excess methanol was then distilled off and the remaining ester subjected to the usual aminolysis previously described. In both cases the solid amide was formed, thus showing that sufficient methanolysis had taken place to

render the ester aminolyzable.

N-Benzylamides from Acids.

The preparation of acetanilide by heating aniline and acetic acid together is a common laboratory experiment. It was thought that N-benzylamides might be prepared in a similar manner by heating benzylamine with free acids. To test this deduction 1 ml. each of isocaproic acid and pimelic acid were heated separately with benzylamine using 0.1 gram of  $\text{NH}_4\text{Cl}$  in each run. After one hour's heating at the boiling point of benzylamine, the mixtures were cooled, and washed with 5 percent KOH solution. In both cases the N-benzylamides were obtained in good yield.



## Results

Thirty-six esters were converted to N-benzylamides in this research, twenty-two of which proved to be new compounds. These latter were analyzed for nitrogen by the Kjeldahl method to establish identity and purity.

Following is a table listing the amides prepared, their melting points, the solvents used for their recrystallization, and analytical data for the new compounds.

## N-BENZYLAMIDE DERIVATIVES FOR ESTERS

N-Benzylamides	Formula	% Nitrogen		Recrystal- lized from	Melting Point °C.	
		Calc.	Found		Found	Lit. Value
n-Valeric	-	-	-	A	42-3	41.1-1.8*
Propionic	-	-	-	A	42-3	42.6-3.7*
n-Caproic	-	-	-	A	52-3	50.3-1.4*
Acetic	-	-	-	A	60-1	60.7-1.3*
Lauric	$C_{19}H_{41}ON$	4.84	4.88	B	83-4	-
Isobutyric	-	-	-	A	86.5-7.5	87-8*
Stearic	$C_{25}H_{43}ON$	3.75	3.89	B	87-8	-
p-Amino- benzoic	$C_{14}H_{14}ON_2$	12.39	12.46	A	89-90	-
Palmitic	$C_{23}H_{39}ON$	4.06	4.11	B	91-2	-
Trichloro- acetic	-	-	-	B	93-4	93.6-4.4*
m-Nitro- benzoic	-	-	-	B	100-1	101**
Glycolic	$C_9H_{11}O_2N$	5.28	5.30	B	103-4	-
Benzoic	-	-	-	A	105-5.5	105-7**
2-Furoic	$C_{12}H_{11}O_2N$	6.96	7.23	A	111-11.5	-
Phenylacetic	-	-	-	B	121-2	122**
Anthranilic	$C_{14}H_{14}ON_2$	12.39	12.58	A	122-3.5	-
Anisic	-	-	-	A	132-3.5	131**
n-Butyl- malonic	$C_{21}H_{26}O_2N_2$	8.28	8.39	C	134-6	-
Salicylic	-	-	-	C	136.5-7	137**
Ethylmalonic	$C_{19}H_{22}O_2N_2$	9.03	9.18	C	137-8.5	-
3-(2-Furyl)- acrylic <sup>1</sup>	-	-	-	A	137-40	-
m-Hydroxy- benzoic	$C_{14}H_{15}O_2N$	6.16	6.08	D	140-1.5	-
p-Nitrobenzoic	-	-	-	B	140.5-2	141-3**
Malonic	-	-	-	C	141.5-2.5	142***

N-Benzylamides	Formula	% Nitrogen		Recrystal- lized from	Melting Point °C.	
		Calc.	Found		Found	Lit. Value
Maleic	$C_{18}H_{18}O_2N_2$	9.51	9.58	C	150.5-1.5	-
Pimelic	$C_{21}H_{26}O_2N_2$	8.23	8.36	C	151-2.5	-
Sebacic	$C_{24}H_{32}O_2N_2$	7.36	7.34	C	160-2	-
Phenylethyl- malonic	$C_{25}H_{26}O_2N_2$	7.25	7.37	C	163-5	-
Carbanic	-	-	-	B	166-7.5	168**
Citric	$C_{27}H_{29}O_4N_3$	9.15	9.14	B	169-71	-
Phthalic	-	-	-	C	178-9	178-9**
Adipic	$C_{20}H_{24}O_2N_2$	8.64	8.61	C	187.5-8.5	-
Tartaric	-	-	-	E	197-200	199**
Fumaric	$C_{18}H_{18}O_2N_2$	9.51	9.58	E	203-4.5	-
Cinnamic	$C_{16}H_{15}ON$	5.90	6.05	E	204-7	-
Oxalic	-	-	-	A	223-4	218**

1. No nitrogen analysis made owing to insufficient quantity of compound.

A, acetone-water

B, acetone

C, isopropyl alcohol

D, isopropyl alcohol-water

E, nitrobenzene

\* Buchler and Mackenzie, J. Am. Chem. Soc., 59, 421 (1937)

\*\* Prager, Jacobson, Schmidt, and Stern, "Beilsteins Handbuch der organischen Chemie", Vol. 12, Julius Springer, Berlin, 4th edition, 1929

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### Failures of Method.

Methyl levulinate and ethyl o-benzoylbenzoate gave only gummy products when subjected to aminolysis with benzylamine.

The N-benzylamides of oleic acid and isocaproic acid were unsatisfactory as derivatives for identification because of their low melting points. Both melted at about 25° (An exact determination was not made.).

Ethyl carbonate resisted all attempts to aminolyse it. This is contrary to the reactivity exhibited by other dibasic esters in this study, but agrees with the known difficulty of hydrolyzing alkyl carbonates.

When added to benzylamine, methyl p-toluenesulfonate gave a water-soluble white solid, no doubt N-methyl-N-benzylammonium p-toluenesulfonate, which was not investigated. Refluxing this solid in benzylamine as usual produced a water-insoluble solid whose melting point did not correspond with the literature value for N-benzyl-p-toluenesulfonamide. This was the only sulfonic ester tried, so no general conclusions concerning the behavior of such esters in aminolysis can be drawn.

## DISCUSSION OF RESULTS

The method of preparing derivatives for identification of the acyl group in esters developed and used in this work seems to present several advantages over other methods now in use. It requires but a single reaction consuming not more than one hour's time, employs only one reagent and is applicable to practically all types of esters. It has particular advantages in dealing with esters of hydroxy-acids such as glycolic and tartaric, which cannot be converted to acid chlorides as is commonly done in the standard procedure.

The method of Koelsch and Tenenbaum (3) and Hardy (4) requires the use of a Grignard reagent which must be prepared each time it is needed because of its instability. Although their method requires only five minutes refluxing time (after the reagent has been prepared), Koelsch and Tenenbaum find that it fails for several common esters of dibasic acids. Our method has been found to work exceptionally well for dibasic esters.

Three principal improvements in Buehler and Mackenzie's (1) work have been accomplished during the course of this investigation.

It has been proved that the use of water as a solvent in the aminolysis of esters by benzylamine decreases the reaction velocity. This decrease can be explained on the basis of dilution of the reactants as well as the impossibility of attaining a high reaction temperature due to preferential refluxing of the water. This latter factor is probably the reason why Buehler and Mackenzie failed to obtain satisfactory yields with esters of the long-chain aliphatic acids. It was found in our own study that the long-chain fatty acid esters gave even better

yields than the shorter-chain compounds.

The use of ammonium chloride as a catalyst has been proved to approximately double the yield of amide in a one hour period with ethyl benzoate. This improvement in yield, which is believed to be general, is obviously an advantage over Buehler and Mackenzie's method since it brings within scope many esters which would otherwise require an inconveniently long period of time for aminolysis.

The conversion of esters of longer-chain alcohols to methyl esters by methanolysis, so that they may be aminolyzed, constitutes a satisfactory method of dealing with an otherwise difficult class of esters. The method of identification set forth in this work is thereby extended, and this extension may certainly be classed as an improvement over Buehler and Mackenzie's method.

The list of N-benzylamides prepared by the above workers has been extended to include a greater variety of esters, thus making it much more useful to the organic analyst.

Inspection of the table of derivatives reveals that although "bunching" of the melting points occurs in a few places, a very good scattering is presented as a whole.

The principal limitation of the method encountered in our work was its failure to give crystalline derivatives with esters of keto-acids. The condensation of benzylamine with acetoacetic ester (27) and with aromatic ketones (28) has been studied, and found to be usually of the type:



Although no investigation of the reason for failure of keto-esters to give satisfactory derivatives was made in our work, it is believed highly probable that a condensation reaction of the sort illustrated above takes place giving compounds which do not respond to the ordinary methods of purification.

The preparation of N-benzylisocaproamide and N,N'-dibenzylpimelamide from the free acids shows that aminolysis with benzylamine may also find use as a method for obtaining N-benzylamides from acids without the intermediate preparation of the acid chlorides. In addition, it appears probable that such derivatives may be prepared from unsubstituted amides. The only example of this reaction in the present study was the preparation of N,N'-dibenzylurea from ethyl carbamate. The reaction has been employed by Clark (29) to prepare derivatives of simple amides by heating them with aniline or alpha-naphthylamine, but benzylamine should certainly prove superior.

The preparation of a N-benzylamide gives almost no information as to the identity of the alkoxyl group present in an ester, although if one has also a knowledge of the physical constants of the ester, the number of such groups possible is extremely limited.

A worthwhile research parallel to the present one would be the development of a method for obtaining derivatives of the alkoxyl group in an ester by a single step.

## SUMMARY

An improved procedure for the preparation of N-benzylamides as derivatives for identifying the acyl group in esters has been developed, and tested on forty-two compounds. It has proved applicable to all classes of carboxylic esters studied except those of keto-acids and carbonic acid.

The advantages of using benzylamine to effect the aminolysis, of omitting water as a solvent, and of using ammonium chloride as a catalyst have been demonstrated.

A table showing the analytical data and physical properties for the N-benzylamide derivatives of thirty-six esters is presented.

Twenty-two new compounds have been synthesized, analyzed for nitrogen, and their melting points recorded.



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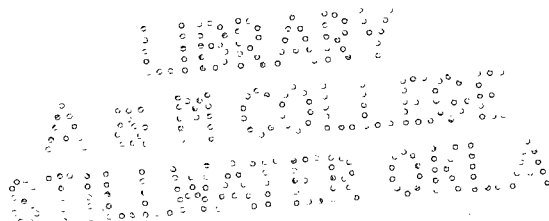
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### BIOGRAPHY

Jack Ardis King was born at Wheatland, Oklahoma, November 30, 1917. He received his grade school training in Shawnee, Oklahoma, and was graduated from the Shawnee High School in 1934.

In 1936, he entered Oklahoma A. and M. College, and was graduated from that institution in June, 1940, with the Bachelor of Science Degree in Chemistry.

In September, 1940, he was enrolled in the graduate school of Oklahoma A. and M. College, where he has been employed as a graduate assistant in the Department of Chemistry until the present time.



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