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# Undergraduate Research Has a Long and Distinguished History at the University of Kentucky

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### Undergraduate research has a long and distinguished history at the University of Kentucky.

**O**n October 14, 2004, William N. Lipscomb, UK's only living Nobel Laureate, delivered the Blazer Lecture, entitled "The Pursuit of Ideas in Science." In his talk, Professor Lipscomb presented a particularly strong endorsement of undergraduate research. He described his own undergraduate research experience at UK and the circumstances under which he performed it and wrote his first publication. "I finished early the laboratory work in a course, 'Qualitative Organic Chemistry.' I then met with Prof. [Robert H.] Baker to see if we could organize an undergraduate research study. Hence this: my first publication! It was published in the Journal of the American Chemical Society."

Professor Lipscomb has graciously agreed to the re-publication of his undergraduate research in Kaleidoscope. What follows is his autobiography, written at the time he received the Nobel Prize for Chemistry in 1976.

#### Autobiography at the time of the Nobel Prize

Although born in Cleveland, Ohio, USA, on December 9, 1919, I moved to Kentucky in 1920, and lived in Lexington through my university years. After my bachelors degree at the University of Kentucky, I entered graduate school at the California Institute of Technology in 1941, at first in physics. Under the influence of Linus Pauling, I returned to chemistry in early 1942. From then until the end of 1945, I was involved in research and development related to the war. After completion of the Ph.D., I joined the faculty of the University of Minnesota in 1946, and moved to Harvard University in 1959. Harvard's recognitions include the Abbott and James Lawrence Professorship in 1971, and the George Ledlie Prize in 1971.

The early research in borane chemistry is best summarized in my book *Boron Hydrides* (W.A. Benjamin, Inc., 1963), although most of this and late work is in several scientific journals. Since about 1960, my research interests have also been concerned with the relationship between three-dimensional structures of enzymes and how they catalyze reactions or how they are regulated by allosteric transformations.

Besides memberships in various scientific societies, I have received the Bausch and Lomb honorary science award in 1937; and, from the American Chemical Society, the Award for Distinguished Service in the Advancement of Inorganic Chemistry, and the Peter Debye Award in Physical Chemistry. Local sections of this Society have given the Harrison Howe Award and Remsen Award. The University of Kentucky presented to me the Sullivan Medallion in 1941, the Distinguished Alumni Centennial Award in 1965, and an honorary Doctor of Science degree in 1963. A Doctor Honoris Causa was awarded by the University of Munich in 1976. I am a member of the National Academy of Sciences U.S.A. and of the American Academy of Arts and Sciences, and a foreign member of the Royal Netherlands Academy of Sciences and Letters.

My other activities include tennis and classical chamber music as a performing clarinetist.

From *Les Prix Nobel en 1976*, Editor Wilhelm Odelberg, [Nobel Foundation], Stockholm, 1977.

This autobiography/biography was written at the time of the award and later published in the book series *Les Prix Nobel/Nobel Lectures*. The information is sometimes updated with an addendum submitted by the Laureate.

To read Prof. Lipscomb's Nobel Lecture, go to <http://nobelprize.org/chemistry/laureates/1976/lipscomb-lecture.pdf>

For general information about all aspects of Prof. Lipscomb's Nobel Prize, go to <http://nobelprize.org/chemistry/laureates/1976/>



**Mentor:**  
Dr. Robert Baker  
Professor, Department of Chemistry



Prof. Lipscomb delivering a lecture recently.

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## The Identification of Alcohols in Aqueous Solution

By WILLIAM N. LIPSCOMB AND ROBERT H. BAKER<sup>1</sup>

The identification of an alcohol when it appears in aqueous solution as from the saponification of an ester usually involves its isolation by repeated distillation, salting out, and drying. Henstock<sup>2</sup> was able to avoid this time-consuming procedure by the use of the Schotten-Baumann reaction of *p*-nitrobenzoyl chloride at  $-15^{\circ}$ .

We have found that the more satisfactory 3,5-dinitrobenzoates may be formed by slight modification of the Henstock procedure. The process involves adding alkali and sodium acetate to the aqueous solution of the alcohol and shaking it at  $0^{\circ}$  with a hydrocarbon solution of 3,5-dinitrobenzoyl chloride.

The yield of ester is affected rather critically by the temperature, the nature and concentration of the alkali, and to a lesser extent by the catalytic effect of sodium acetate. Preliminary experiments showed that it was necessary to dissolve the solid dinitrobenzoyl chloride in an inert solvent so as to keep it in a liquid phase during the course of the reaction. When ether is used for this purpose the melting points of the esters, with the exception of ethyl, are consistently low. This is true even when extreme care is taken to remove all alcohol from the ether and therefore must be due to cleavage of the ether. Benzene is satisfactory except for its high melting point and this is easily lowered by the addition of dry ligroin.

Although the yield of ethyl ester is greater at  $0^{\circ}$  than at room temperature, it is not necessary to employ the difficultly-maintained lower temperatures. The yield of ethyl ester is four times as great when formed in the presence of 5% sodium hydroxide as it is when a similar concentration of sodium carbonate is used, and weaker bases such as sodium bicarbonate give negligible yields of the ester. Increasing the concentration of sodium hydroxide in the reaction mixture at  $0^{\circ}$  increased the yield of ethyl ester as shown in Fig. 1. It is impractical to use concentrations of sodium hydroxide in excess of 20% because the reaction mixture becomes semi-solid and difficult to handle.

(1) Present address, Northwestern University, Evanston, Illinois.  
(2) Henstock, *J. Chem. Soc.*, 216 (1933).

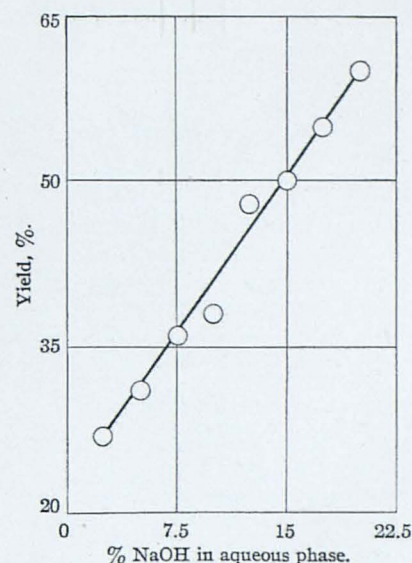


Fig. 1.

**Reagent.**—To 25 g. of 3,5-dinitrobenzoyl chloride is added 50 ml. of dry sulfuric acid-washed ligroin and the resulting solution is then diluted to 250 ml. with dry benzene.

**Procedure.**—One-half gram of sodium acetate crystals is dissolved in 10 ml. of a 5% aqueous solution of the alcohol, then 10 ml. of 40% sodium hydroxide is added and the solution cooled to  $0^{\circ}$ . Five ml. of the solution of 3,5-dinitrobenzoyl chloride in benzene-ligroin is then added and the mixture alternately shaken and placed in an ice-bath for one-half hour. The mixture is then transferred to a separatory funnel and extracted with 30 ml. of ether. The ether solution is washed with an equal volume of water, then 5% hydrochloric acid, and again with water. Evaporation of the solvent yields the crude derivative as recorded in Table I.

TABLE I

Alcohol	Crude ester, g.	Yield, %	M. p. of crude ester, $^{\circ}$ C.	M. p. in lit., $^{\circ}$ C.
Methyl	0.09 <sup>b</sup>	17	94-96	107
Ethyl	.27	52	90-91	93
<i>n</i> -Propyl	.37	67	72-73	74
Isopropyl	.10	18	117-118	122
<i>n</i> -Butyl	.37	64	62-63	64
<i>s</i> -Butyl	.06	10	72-73	75
Isobutyl	.30	52	83-84	86
<i>t</i> -Butyl	.01	1		142
Allyl	.22	42	40-41	48

<sup>a</sup> Shriner and Fuson, "Systematic Identification of Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 2nd Ed., 1940, p. 185. <sup>b</sup> Obtained only after three successive additions and shakings with the acid chloride solution.

CONTRIBUTION FROM THE  
CHEMICAL LABORATORY  
UNIVERSITY OF KENTUCKY RECEIVED OCTOBER 6, 1941