values for Folin color per 10 μg. N are given in Table I. Within experimental error antipneumococcal antibodies and human gamma globulin give identical results.

TABLE I

COLOR EQUIVALENTS OF HUMAN ANTIBODY AND NORMAL HUMAN GAMMA GLOBULIN

	Hexosamine[a,b]/Total N	Methylpentose[b]/Total N	Folin-Ciocalteu color $\frac{D\ 7500}{10\ \mu g.\ N}$
Anti-C		0.032[e]	
Anti-SII	0.07[c]		0.155
Anti-SIII	.07[d]	.030[f]	.150
Gamma globulin			
Fraction II-1, 2[13]	.07		.149
Fraction II-3[13]	.07	.034	.146
Sample B[13]		.032	
Sample S[13]		.026	

[a] Hexosamine was determined after hydrolysis with 2 *N* HCl at 100° for 2 hours. [b] The color values, while reproducible, are not assumed to be specific for either hexosamine or methylpentose.[15,16] [c] Correction for hexosamine color value of SII-14%. [d] Correction for hexosamine color value of SIII-7%. [e] Correction for methylpentose color value of "C" substance-8%. [f] Correction for methylpentose color value of SIII-14%.

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[15] E. Vasseur and J. Immers, *Arkiv Kemi*, **1**, 253 (1949).

[16] H. N. Horowitz, M. Ikawa and M. Fling, *Arch. Biochem.*, **25**, 226 (1950).

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The Attempted Rosenmund Reduction of Diphenylacetyl Chloride[1]

BY JOHN G. BURR, JR.

Recently a supply of diphenylacetaldehyde was desired in this Laboratory, and since diphenylacetyl chloride was available, the Rosenmund reduction of this compound was investigated. The products which were obtained are

$$(C_6H_5)_2CHCOCl \longrightarrow \underset{47\%}{(C_6H_5)_2CHCH(C_6H_5)_2} + \underset{13.5\%}{(C_6H_5)_2CH_2} + \underset{13.5\%}{(C_6H_5)_2CHCOOH} + \underset{75\%}{HCl}$$

The occurrence of a product of the nature of tetraphenylethane as a product of a Rosenmund reduction has not been previously reported. Triphenylacetyl chloride also loses carbon monoxide and hydrogen chloride under Rosenmund conditions; the product in this case is triphenylmethane.[2]

The more usual products and by-products of this reaction were accounted for by Rosenmund[3] as

$$\begin{tikzcd}
 & RCOCH_2R \arrow[r] & RCH_3 + RCOOH + RCH_2CH_2R & \\
R{-}COCl \arrow[ur] \arrow[dr] & & & \\
 & RCHO \arrow[r] & RCH_2OH \arrow[r] & (RCH_2)_2O
\end{tikzcd}$$

This scheme does not predict products of the nature of tetraphenylethane or triphenylmethane.

A possible explanation of these two products may be found in the temperature at which these reactions were carried out. It is known, for example, that diphenylacetyl chloride decomposes at temperatures of around 200–250° into carbon monoxide, hydrogen chloride, tetraphenylethylene and diphenylketene.[4,5] Staudinger[4] explains the formation of tetraphenylethylene through an intermediate diphenylchloromethane which is known to decompose upon heating to tetraphenylethylene[6,7] and tetraphenylethane.[7] The moderate

$$(C_6H_5)_2CHCOCl \longrightarrow [(C_6H_5)_2CHCl] + CO$$

$$[(C_6H_5)_2CHCl] \longrightarrow (C_6H_5)_2C{=}C(C_6H_5)_2 + (C_6H_5)_2CHCH(C_6H_5)_2 + HCl$$

temperature of the reaction observed here (boiling xylene solution) with its long duration might well be sufficient for a smooth production of diphenylchloromethane (but little diphenylketene), and the thermal conversion of this to tetraphenylethylene and tetraphenylethane. The tetraphenylethylene, in the reductive atmosphere, might be converted to tetraphenylethane.

Similarly, triphenylacetylchloride is known[5] to be converted at 170–180° quantitatively to triphenylchloromethane and carbon monoxide. Under the conditions of the Rosenmund reduction, the triphenylchloromethane would probably be converted to triphenylmethane.

Experimental[8]

Attempted Rosenmund Reduction of Diphenylacetyl Chloride.—A suspension of 4 g. of 5% palladium–barium sulfate catalyst, poisoned with 0.6 ml. of quinoline-S solution, in 200 ml. of toluene was prepared, heated to boiling, and a small amount of solvent distilled to dry the remainder. To the cooled solution was added 44.6 g. of diphenylacetyl chloride. A moderately fast stream of hydrogen was passed through the stirred, refluxing mixture. The effluent gases were passed into water, and the absorbed hydrogen chloride titrated with 5 *N* sodium hydroxide. After overnight reaction, 28 ml. of NaOH had been consumed (75% of theory). The cooled solution was filtered from the catalyst, and the solvent was removed under vacuum. The residual pasty solid was heated with aqueous sodium bicarbonate. The undissolved material was filtered off, and the filtrate, after extraction with ether, acidified to give 5.5 g. (13.5%) of diphenylacetic acid (m.p. 145°). The neutral organic substances were stirred with ether. The ether-soluble material was obtained by filtration. It was evaporated, and the residue distilled at high vacuum. The distillate, 4.4 g. (13.5%), b.p. 87–89° (0.1 mm.), $n^{25}D$ 1.5788, was diphenylmethane. The residue was about 5 g. of a semisolid.

The ether-insoluble material weighed 15 g. (47% yield), and after several crystallizations from benzene formed a white microcrystalline powder melting at 211–212°. This material conforms in analysis, melting point, and general solubilities to tetraphenylethane. A mixture melting point with authentic tetraphenylethane (prepared from diphenylchloromethane and zinc, and melting at 211–212°) showed no depression.

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[1] This document is based upon work performed under Contract Number W-7405, eng. 26 for the Atomic Energy Commission at the Oak Ridge National Laboratory.

[2] S. Daniloff and E. Venus-Danilova, *Ber.*, **59**, 377 (1926).

[3] K. W. Rosenmund and F. Zetzche, *ibid.*, **54**, 642, 2038 (1921).

[4] H. Staudinger, *ibid.*, **44**, 1619 (1911).

[5] A. Bistrzycki and A. Landtwing, *ibid.*, **41**, 686 (1908).

[6] R. Anschütz, *Ann.*, **235**, 220 (1886).

[7] C. Engler and H. Bethge, *Ber.*, **7**, 1128 (1886).

[8] All melting points were taken on a Fisher-Johns block and are uncorrected. Microanalyses are by Dr. H. W. Galbraith, Knoxville, Tenn.

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Phenylalanine Analogs

BY J. H. BURCKHALTER AND VERLIN C. STEPHENS

The following new compounds related to phenyl-