3. A mechanism is suggested for the formation of OH during the photolysis of formic acid. This mechanism is consistent with the mechanism previously suggested for acetic acid.

4. A preliminary experiment with acetaldehyde showed HCO to be a stable radical at room temperature.

5. In view of the difficulty in reconciling the results of Gorin and Taylor with those of Terenin, it appears probable that the major portion of the photolysis of formic acid takes place by decomposition into stable molecules in one primary act.

UNIVERSITY HEIGHTS
NEW YORK, N. Y. RECEIVED JULY 9, 1936

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF MONTANA STATE UNIVERSITY]

Cyclohexyltrichloromethylcarbinol

BY JOSEPH W. HOWARD AND ROBERT J. BROWN

This compound was prepared by the reaction of chloral and cyclohexylmagnesium bromide. The most satisfactory results were obtained when this was carried out as an "inverse Grignard" reaction.

The Grignard reagent was prepared by adding 82 g. of monobromocyclohexane in 60 cc. of ether to 14 g. of magnesium ribbon and a crystal of iodine in 70 cc. of ether. The reaction was completed after three hours of stirring and gentle heating. The resultant solution was decanted from any unchanged magnesium and added with stirring over a two-hour period to 74 g. of freshly distilled chloral in 150 cc. of ether. The product was decomposed with 30% sulfuric acid and the ether washed in turn with water, sodium bicarbonate solution, sodium bisulfite solution and water. It was dehydrated over anhydrous sodium sulfate, the ether removed by distillation and the alcohol distilled in vacuum. A yield of 35 g. of an oily liquid boiling at 119 to 121° at 15 mm. was obtained; d^{20}_{20} 1.2839, n_{25} 1.4820. *Anal.* Calcd. for $C_8H_{13}OCl_3$: Cl, 45.95. Found: Cl, 45.99.

This carbinol darkens on standing. It is insoluble in water but soluble in ether, ethyl alcohol, methyl alcohol, acetone, benzene, chloroform and carbon tetrachloride.

Preparation of the Esters

The acetate and propionate were prepared by refluxing the carbinol for one and one-half hours at 135° with the corresponding acid chloride. The same method was used to prepare the butyrate but butyric anhydride was found to give more satisfactory results than butyryl chloride. The benzoate was prepared by treatment with benzoyl chloride according to the usual procedure of the Schotten–Baumann reaction.

ESTERS OF CYCLOHEXYLTRICHLOROMETHYLCARBINOL

		Formula	Analysis for Cl, % Calcd.	Found
1	Acetate	$C_{10}H_{15}O_2Cl_3$	38.89	39.08
2	Propionate	$C_{11}H_{17}O_2Cl_3$	36.97	37.14
3	Butyrate	$C_{12}H_{19}O_2Cl_3$	35.44	35.28
4	Benzoate	$C_{15}H_{17}O_2Cl_3$	31.71	31.56

	B. p., °C.	Mm.	d^{20}_{20}	n_{25}
1	173	680	1.3612	1.4945
2	188	681.6	1.2119	1.4989
3	185	682.6	1.1872	1.4995
4	210	683.3	1.2893	1.5259

The average yields of these esters was 85%. They are all insoluble in water but soluble in ether, ethyl alcohol, methyl alcohol, acetone, benzene, chloroform and carbon tetrachloride.

Summary

Cyclohexyltrichloromethylcarbinol as well as its acetic, propionic, butyric and benzoic esters have been prepared and studied.

MISSOULA, MONTANA RECEIVED JUNE 15, 1936