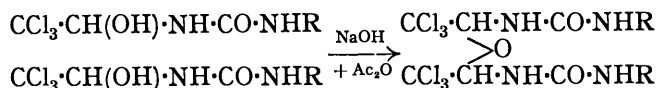


30. *The Condensation of Chloral and Bromal with Diamides.*

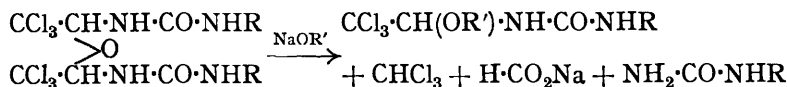
By F. D. CHATTAWAY and E. J. F. JAMES.

WITH mono- and unsymmetrical di-alkyl- and -aryl-ureas chloral condenses as readily as with urea itself (Chattaway and James, *Proc. Roy. Soc.*, 1931, *A*, **134**, 372), though with such substituted compounds only the first stage of the reaction can occur. Thus methyl- and ethyl-urea in aqueous solution yield with chloral *N*-methyl- and *N*-ethyl-*N'*-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)urea respectively, the NHR group showing no tendency to react further. The products are colourless substances, which with acetic anhydride form *diacetyl* derivatives, $\text{CCl}_3\cdot\text{CH}(\text{OAc})\cdot\text{NH}\cdot\text{CO}\cdot\text{NACR}$. They dissolve readily in aqueous alkalis, and

addition of acetic anhydride to the alkaline solutions produces *bis*-($\beta\beta\beta$ -trichloro- α -N-alkylcarbamidoethyl) ethers, which themselves yield *diacetyl* derivatives.



When any one of these ethers is kept for several hours with a solution of any sodium alkoxide in the corresponding alcohol, fission occurs at the ether linkage and an N-alkyl-N'-($\beta\beta\beta$ -trichloro- α -alkoxyethyl)urea is formed.



Bromal condenses similarly with monosubstituted ureas, but the reactions do not proceed as readily as with chloral and the resulting compounds are somewhat less stable.

The condensation of chloral with unsymmetrical dimethyl- and diethyl-urea was first observed by van der Zande (*Rec. trav. chim.*, 1889, **8**, 222), who found that the compound produced from dimethylurea separated from water in a monohydrated condition, $\text{CCl}_3\cdot\text{CH}(\text{OH})\cdot\text{NH}\cdot\text{CO}\cdot\text{NMe}_2\cdot\text{H}_2\text{O}$, the molecule of water being lost on keeping or warming, whereas the similar condensation product from diethylurea formed anhydrous crystals. Bromal condenses similarly with dimethylurea in aqueous solution, the condensation product separating first as an unstable *hydrate*, which on keeping loses water, yielding the anhydrous compound.

Chloral and bromal also condense with unsymmetrical diphenylurea, the compounds $\text{CX}_3\cdot\text{CH}(\text{OH})\cdot\text{NH}\cdot\text{CO}\cdot\text{NPh}_2$ (X = Cl or Br) being formed.

These condensation products of chloral and bromal with unsymmetrical dialkyl- and diaryl-ureas all dissolve in dilute aqueous alkalis, but separate unchanged on addition of acetic anhydride, ethers not being formed.

Chloral condenses not only with urea and substituted ureas, but with other diamides, e.g., oxamide and malonamide. Oxamide, on account of its sparing solubility, only very slowly condenses, and it is necessary to heat it with anhydrous chloral for many hours to obtain any quantity of the *dichloraloxamide* [*NN'*-*bis*-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)oxamide], $\text{CCl}_3\cdot\text{CH}(\text{OH})\cdot\text{NH}\cdot\text{CO}\cdot\text{CO}\cdot\text{NH}\cdot\text{CH}(\text{OH})\cdot\text{CCl}_3$, which is formed. It has not been found possible to isolate monochloraloxamide, as, on account of the very sparing solubility of oxamide, chloral is always necessarily present in excess and the monochloraloxamide is itself further, and preferentially, acted upon.

The action of ammonia upon chloraloxamethane [ethyl N-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)oxamate], $\text{CCl}_3\cdot\text{CH}(\text{OH})\cdot\text{NH}\cdot\text{CO}\cdot\text{CO}_2\text{Et}$, which is readily formed by the condensation of chloral with oxamethane, likewise fails to produce monochloraloxamide, as the ammonia causes hydrolysis of the chloraloxamethane, with separation of chloroform. The action of aniline upon chloraloxamethane, however, produces the phenyl derivative of monochloraloxamide, viz., N-*phenyl*-N'-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)oxamide, $\text{CCl}_3\cdot\text{CH}(\text{OH})\cdot\text{NH}\cdot\text{CO}\cdot\text{CO}\cdot\text{NHPh}$.

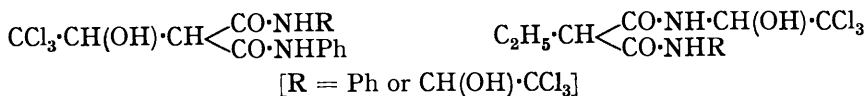
The chloral is very loosely attached to the amide molecule both in dichloraloxamide and in chloralphenyloxamide. The compounds are slowly decomposed into chloral and the amide when they are heated above 150° for any length of time; they are also very rapidly broken down by dilute alkalis. Some oxamide is even formed when they are dissolved in aqueous alcohol.

Dichloraloxamide is a well-crystallised substance, which with acetic anhydride forms a *diacetyl* derivative. It reacts readily with phosphorus pentachloride, with formation of *NN'*-*bis*-($\alpha\beta\beta\beta$ -tetrachloroethyl)oxamide. The two chlorine atoms in the α -positions in this compound are very reactive, being displaced by ethoxy-, amino-, and anilino-groups by the action of alcohol, ammonia, and aniline, respectively, upon it.

Malonamide also condenses with chloral, the reaction here being more complicated on account of the reactivity of the methylene group. When malonamide is heated for several

hours with anhydrous chloral, $\text{CNN}'\text{-tris}(\beta\beta\text{-trichloro-}\alpha\text{-hydroxyethyl})\text{malonamide}$, $\text{CCl}_3\text{-CH(OH)\cdot CH[CO-NH-CH(OH)\cdot CCl}_3\text{]}_2$, is formed.

When a hydrogen atom, either in one of the amido-groups or in the methylene group in malonamide, is replaced by an alkyl or an aryl radical, condensation only occurs with the unsubstituted group or groups. Thus by condensing chloral with the appropriate substituted malonamide, the following compounds have been obtained :



When chloral is condensed with malonanilic acid, carbon dioxide is eliminated and $\gamma\gamma\gamma\text{-trichloro-}\beta\text{-hydroxybutyranilide}$ is formed.

All the condensation products of chloral with malonamides are colourless crystalline substances, which yield *mono-*, *di-*, or *tri-acetyl* derivatives, according to the number of hydroxy-groups present in the molecule.

Although the methylene group of malonamide is sufficiently reactive to condense with chloral, that of cyanoacetamide is not, only the amido-group reacting, with formation of *cyanoaceto-}\beta\beta\beta\text{-trichloro-}\alpha\text{-hydroxyethylamide}*, $\text{CCl}_3\text{-CH(OH)\cdot NH}\cdot\text{CO}\cdot\text{CH}_2\text{-CN}$.

EXPERIMENTAL.

The Condensation of Chloral and Bromal with Substituted Ureas.—(1) When a solution of 5 g. of methylurea and 20 g. (2 mols.) of chloral hydrate in 20 c.c. of water was kept at the ordinary temperature, *N-methyl-N'-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-hydroxyethyl})\text{urea}* slowly crystallised. It separated from aqueous alcohol in lustrous six-sided plates, m. p. 140° (decomp.) (Found : Cl, 47.7. $\text{C}_4\text{H}_7\text{O}_2\text{N}_2\text{Cl}_3$ requires Cl, 48.0%), and its *diacetyl* derivative from aqueous acetic acid in long, slender, colourless, flattened prisms, m. p. 90—91° (decomp.) (Found : Cl, 35.1. $\text{C}_8\text{H}_{11}\text{O}_4\text{N}_2\text{Cl}_3$ requires Cl, 34.8%).

(2) 5 G. of *N-methyl-N'-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-hydroxyethyl})\text{urea}* were dissolved in 45 c.c. of ice-cold *N-sodium hydroxide* (2 mols.) and 2.4 g. of acetic anhydride were added drop by drop, with shaking. *Bis-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-N'-methylcarbamidoethyl})\text{ ether}* separated immediately as a colourless flocculent solid. It crystallised from boiling alcohol in long slender prisms, m. p. 230° (decomp.) (Found : Cl, 49.9. $\text{C}_8\text{H}_{12}\text{O}_3\text{N}_4\text{Cl}_6$ requires Cl, 50.0%). Its *diacetyl* derivative crystallised from dilute acetic acid in elongated colourless plates, m. p. 131° (decomp.) (Found : Cl, 42.0. $\text{C}_{12}\text{H}_{16}\text{O}_5\text{N}_4\text{Cl}_6$ requires Cl, 41.8%).

(3) 3 G. of the preceding ether were dissolved in 30 c.c. of alcohol, and a solution of 0.5 g. of sodium in 30 c.c. of alcohol was added. After 24 hours, the liquid, which smelt strongly of chloroform, was poured into 200 c.c. of water; *N-methyl-N'-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-ethoxyethyl})\text{urea}* then crystallised. It separated from alcohol in long, slender, colourless prisms, m. p. 145° (decomp.) (Found : Cl, 43.1. $\text{C}_6\text{H}_{11}\text{O}_2\text{N}_2\text{Cl}_3$ requires Cl, 42.7%).

By similar reactions the following compounds were prepared :

N-Ethyl-N'-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-hydroxyethyl})\text{urea}, large, four-sided, colourless tablets, from water, m. p. 138° (decomp.) (Found : Cl, 45.3. $\text{C}_5\text{H}_9\text{O}_2\text{N}_2\text{Cl}_3$ requires Cl, 45.2%).

N-Acetyl-N-ethyl-N'-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-acetoxethyl})\text{urea}, long, colourless, flattened prisms, with domed ends, from aqueous acetic acid, m. p. 60° (decomp.) (Found : Cl, 33.2. $\text{C}_9\text{H}_{13}\text{O}_4\text{N}_2\text{Cl}_3$ requires Cl, 33.7%).

Bis-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-N'-ethylcarbamidoethyl})\text{ ether}, long, slender, colourless, flattened prisms, from alcohol, m. p. 226° (decomp.) (Found : Cl, 47.2. $\text{C}_{10}\text{H}_{16}\text{O}_3\text{N}_4\text{Cl}_6$ requires Cl, 47.0%).

Bis-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-N'-acetyl-N'-ethylcarbamidoethyl})\text{ ether}, long colourless prisms, from acetic acid, m. p. 145° (decomp.) (Found : Cl, 39.3. $\text{C}_{14}\text{H}_{20}\text{O}_5\text{N}_4\text{Cl}_6$ requires Cl, 39.6%).

N-Ethyl-N'-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-methoxyethyl})\text{urea}, long, slender, colourless prisms, from aqueous alcohol, m. p. 150° (decomp.) (Found : Cl, 42.5. $\text{C}_6\text{H}_{11}\text{O}_2\text{N}_2\text{Cl}_3$ requires Cl, 42.7%). The corresponding *ethoxy*-derivative, m. p. 135° (decomp.) (Found : Cl, 40.4. $\text{C}_7\text{H}_{13}\text{O}_2\text{N}_2\text{Cl}_3$ requires Cl, 40.4%), the *n-propoxy*-derivative, m. p. 140° (decomp.) (Found : Cl, 38.35. $\text{C}_8\text{H}_{15}\text{O}_2\text{N}_2\text{Cl}_3$ requires Cl, 38.5%), and the *n-butoxy*-derivative, m. p. 95° (decomp.) (Found : Cl, 36.5. $\text{C}_9\text{H}_{17}\text{O}_2\text{N}_2\text{Cl}_3$ requires Cl, 36.5%), all form long colourless prisms from aqueous alcohol.

NN-Dimethyl-N'-(}\beta\beta\beta\text{-trichloro-}\alpha\text{-hydroxyethyl})\text{urea} separated first as a monohydrate

in very large, colourless, rhombic tablets, m. p. 74° (decomp.), which were prepared for analysis by very rapid drying with filter-paper (Found : Cl, 41.9. Calc. for $C_5H_9O_2N_2Cl_3, H_2O$: Cl, 42.0%). On keeping, water was lost with formation of the normal condensation product, which crystallised from alcohol in colourless flattened prisms, m. p. 157° (decomp.) (van der Zande, *loc. cit.*, gives m. p. 153°) (Found : Cl, 45.0. Calc. for $C_5H_9O_2N_2Cl_3$: Cl, 45.2%).

NN-Diethyl-N'-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)urea, large lustrous leaflets, m. p. 146° (decomp.) (van der Zande gives m. p. 142°) (Found : Cl, 40.4. Calc. for $C_7H_{13}O_2N_2Cl_3$: Cl, 40.4%).

NN-Diphenyl-N'-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)urea, prepared by the action of anhydrous chloral upon diphenylurea, crystallised from alcohol in clusters of long colourless prisms, m. p. 170° (decomp.) (Found : Cl, 29.9. $C_{15}H_{13}O_2N_2Cl_3$ requires Cl, 29.6%). Its monoacetyl derivative formed slender colourless prisms, from acetic acid, m. p. 108° (decomp.) (Found : Cl, 26.6. $C_{17}H_{15}O_3N_2Cl_3$ requires Cl, 26.5%).

The following derivatives of bromal were prepared by similar reactions :

N-Methyl-N'-($\beta\beta\beta$ -tribromo- α -hydroxyethyl)urea, glistening, rectangular, colourless plates, from aqueous alcohol, m. p. 189° (decomp.) (Found : Br, 67.3. $C_4H_7O_2N_2Br_3$ requires Br, 67.6%). The diacetyl derivative crystallised from aqueous acetic acid in lustrous, six-sided, colourless prisms, m. p. 119° (decomp.) (Found : Br, 54.8. $C_8H_{11}O_4N_2Br_3$ requires Br, 54.6%).

Bis-($\beta\beta\beta$ -tribromo- α -N'-methylcarbamidoethyl) ether, colourless compact prisms, from alcohol, m. p. 200° (decomp.) (Found : Br, 68.9. $C_8H_{12}O_3N_4Br_6$ requires Br, 69.3%). The diacetyl derivative crystallised from acetic acid in compact colourless prisms, m. p. 180° (decomp.) (Found : Br, 62.2. $C_{12}H_{16}O_5N_4Br_6$ requires Br, 61.9%).

N-Methyl-N'-($\beta\beta\beta$ -tribromo- α -ethoxyethyl)urea, long, slender, colourless, flattened prisms, from aqueous alcohol, m. p. 162° (decomp.) (Found : Br, 62.4. $C_8H_{11}O_2N_2Br_3$ requires Br, 62.7%).

N-Phenyl-N'-($\beta\beta\beta$ -tribromo- α -hydroxyethyl)urea, prepared by fusing together bromal hydrate (1 mol.) and phenylurea (1 mol.), crystallised from aqueous alcohol in long, slender, colourless, flattened prisms, m. p. 167° (decomp.) (Found : Br, 57.5. $C_9H_9O_2N_2Br_3$ requires Br, 57.6%). The diacetyl derivative crystallised in slender colourless prisms, from acetic acid, m. p. 142° (decomp.) (Found : Br, 47.9. $C_{13}H_{13}O_4N_2Br_3$ requires Br, 48.0%).

Bis-($\beta\beta\beta$ -tribromo- α -N'-phenylcarbamidoethyl) ether, long, slender, colourless prisms, from aqueous alcohol, m. p. 185° (decomp.) (Found : Br, 58.9. $C_{18}H_{16}O_3N_4Br_6$ requires Br, 59.0%). The diacetyl derivative crystallised from acetic acid in colourless rectangular plates, m. p. 163° (decomp.) (Found : Br, 53.4. $C_{22}H_{20}O_5N_4Br_6$ requires Br, 53.4%).

N-Phenyl-N'-($\beta\beta\beta$ -tribromo- α -methoxyethyl)urea, lustrous, irregular, colourless plates, from aqueous alcohol, m. p. 158° (decomp.) (Found : Br, 56.0. $C_{10}H_{11}O_2N_2Br_3$ requires Br, 55.7%). The corresponding ethoxy-compound crystallised from aqueous alcohol in long, slender, colourless prisms, m. p. 145° (decomp.) (Found : Br, 53.9. $C_{11}H_{13}O_2N_2Br_3$ requires Br, 54.0%).

NN-Dimethyl-N'-($\beta\beta\beta$ -tribromo- α -hydroxyethyl)urea, long, slender, colourless prisms, from water, m. p. 165° (decomp.) (Found : Br, 64.3. $C_5H_9O_2N_2Br_3$ requires Br, 65.0%). An unstable hydrate was first formed in large, colourless, rhombic prisms, m. p. 96° (decomp.) (Found : Br, 62.2. $C_5H_9O_2N_2Br_3, H_2O$ requires Br, 62.0%).

NN-Diphenyl-N'-($\beta\beta\beta$ -tribromo- α -hydroxyethyl)urea, prepared by fusing together bromal hydrate (1 mol.) and diphenylurea (1 mol.), crystallised from alcohol in long colourless prisms, m. p. 168° (decomp.) (Found : Br, 49.0. $C_{15}H_{13}O_2N_2Br_3$ requires Br, 48.7%).

The Condensation of Chloral with Oxamide, Malonamide, and Related Compounds.—(1) 10 G. of oxamide and 50 g. of anhydrous chloral (2 mols. + excess) were heated together under reflux for 8 hours. The mixture was then poured into water and the colourless solid was collected, washed with cold water, and extracted several times with boiling alcohol. On addition of water to the alcoholic extract, NN'-bis-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)oxamide crystallised. It separated from aqueous alcohol in colourless flattened prisms (10 g.), which decomposed gradually above 190° into oxamide and chloral (Found : Cl, 55.5. $C_6H_6O_4N_2Cl_6$ requires Cl, 55.6%). Its diacetyl derivative crystallised from acetic acid in clusters of slender colourless prisms, m. p. 192° (Found : Cl, 45.6. $C_{10}H_{10}O_6N_2Cl_6$ requires Cl, 45.6%).

(2) A solution of 5 g. of ethyl N-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)oxamate (prepared by the method of Feist, *Ber.*, 1914, 47, 1173) and 1.8 g. of aniline (1 mol.) in 25 c.c. of alcohol was warmed on the water-bath for 30 minutes. On addition of dilute hydrochloric acid, N-phenyl-N'-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)oxamide separated as a colourless solid. It crystallised from slightly dilute alcohol in flattened prisms, which decomposed above 180° into chloral and N-phenyloxamide (Found : Cl, 33.7. $C_{10}H_9O_3N_2Cl_3$ requires Cl, 34.2%). Its monoacetyl deriv-

ative crystallised from acetic acid in felted prisms, m. p. 185° (Found : Cl, 30.3. $C_{12}H_{11}O_4N_2Cl_3$ requires Cl, 30.1%).

(3) 10 G. of *NN'*-bis-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)oxamide and 10 g. of phosphorus pentachloride (2 mols.) were heated together on the water-bath for 30 minutes. Ice was added to the viscous product, and *NN'*-bis-($\alpha\beta\beta\beta$ -tetrachloroethyl)oxamide then separated as a yellowish solid. It crystallised from acetic acid in lustrous, irregular, colourless plates, m. p. 170° (Found : Cl, 67.2. $C_6H_4O_2N_2Cl_8$ requires Cl, 67.4%).

When crystallised from boiling alcohol, this compound yielded *NN'*-bis-($\beta\beta\beta$ -trichloro- α -ethoxyethyl)oxamide, which separated from alcohol in long colourless prisms, m. p. 176° (Found : Cl, 49.0. $C_{10}H_{14}O_4N_2Cl_6$ requires Cl, 48.5%). The corresponding *diamino*-compound, prepared by warming the octachloro-compound with alcoholic ammonia on the water-bath for 30 minutes, crystallised from boiling alcohol in small, lustrous, four-sided plates, m. p. 213° (Found : Cl, 56.1. $C_6H_8O_2N_4Cl_6$ requires Cl, 55.9%). The corresponding *dianilino*-compound, prepared similarly, crystallised from boiling alcohol in long, slender, colourless, flattened prisms, m. p. 193° (decomp.) (Found : Cl, 39.8. $C_{18}H_{16}O_2N_4Cl_6$ requires Cl, 39.9%).

(4) 5 G. of malonamide and 30 g. (3 mols. + excess) of anhydrous chloral were heated together under reflux on the water-bath for 12 hours. On addition of water to the pale yellow, viscid liquid obtained, and prolonged trituration, *CNN'*-tris-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)-malonamide separated as a colourless flocculent solid. It crystallised from boiling aqueous alcohol in two polymorphic forms : labile form, clusters of long slender prisms, m. p. 180°; stable form, colourless six-sided plates, m. p. 180° (decomp.) (Found : C, 20.05; H, 1.8; N, 5.25; Cl, 58.8. $C_9H_9O_5N_2Cl_9$ requires C, 19.9; H, 1.7; N, 5.1; Cl, 58.7%). Its *triacetyl* derivative crystallised from aqueous acetic acid in colourless slender prisms, m. p. 165° (decomp.) (Found : Cl, 47.4. $C_{15}H_{15}O_8N_2Cl_9$ requires Cl, 47.7%).

By means of similar condensations with the appropriate substituted malonamides the following compounds were obtained :

NN'-*Diphenyl*-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)malonamide, long, slender, colourless prisms, m. p. 187° (decomp.) (Found : Cl, 26.4. $C_{17}H_{15}O_3N_2Cl_3$ requires Cl, 26.5%). Its *acetyl* derivative crystallised from acetic acid in long, slender, colourless prisms, m. p. 210° (decomp.) (Found : Cl, 24.1. $C_{19}H_{17}O_4N_2Cl_3$ requires Cl, 24.0%).

N-Phenyl-N'-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)malonamide, long, slender, colourless prisms, m. p. 170° (decomp.) (Found : Cl, 44.8. $C_{13}H_{12}O_4N_2Cl_6$ requires Cl, 44.8%); *diacetyl* derivative, small colourless prisms, m. p. 175° (decomp.), from alcohol (Found : Cl, 37.8. $C_{17}H_{16}O_6N_2Cl_6$ requires Cl, 38.2%).

NN'-*Bis*-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)ethylmalonamide, clusters of long colourless prisms, m. p. 163° (decomp.), from alcohol (Found : Cl, 50.1. $C_9H_{12}O_4N_2Cl_6$ requires Cl, 50.1%); *diacetyl* derivative, short, compact, colourless prisms, m. p. 132°, from alcohol (Found : Cl, 42.1. $C_{13}H_{16}O_6N_2Cl_6$ requires Cl, 41.9%).

N-Phenyl-N'-($\beta\beta\beta$ -trichloro- α -hydroxyethyl)ethylmalonamide, long colourless prisms, m. p. 164° (decomp.), from aqueous alcohol (Found : Cl, 29.7. $C_{13}H_{15}O_3N_2Cl_3$ requires Cl, 30.1%); *monoacetyl* derivative, clusters of colourless flattened prisms, m. p. 185° (decomp.), from acetic acid (Found : Cl, 27.0. $C_{15}H_{17}O_4N_2Cl_3$ requires Cl, 26.9%).

$\gamma\gamma\gamma$ -*Trichloro- β -hydroxybutyranilide*, prepared by condensing anhydrous chloral with malonanilic acid (carbon dioxide being eliminated), crystallised from aqueous alcohol in glistening, colourless, six-sided leaflets, m. p. 146° (decomp.) (Found : Cl, 37.7. $C_{10}H_{10}O_2NCl_3$ requires Cl, 37.7%). Its *monoacetyl* derivative crystallised from slightly aqueous acetic acid in long, colourless, four-sided prisms, with domed ends, m. p. 149° (decomp.) (Found : Cl, 33.0. $C_{12}H_{12}O_3NCl_3$ requires Cl, 32.8%).

Cyanoaceto- $\beta\beta\beta$ -trichloro- α -hydroxyethylamide, prepared by condensing anhydrous chloral with cyanoacetamide, crystallised from aqueous alcohol in compact, colourless, four-sided prisms, with domed ends, m. p. 155° (decomp.) (Found : Cl, 46.2. $C_5H_5O_2N_2Cl_3$ requires Cl, 46.0%). Its *monoacetyl* derivative crystallised from dilute acetic acid in compact colourless prisms, m. p. 108° (decomp.) (Found : Cl, 39.2. $C_7H_7O_3N_2Cl_3$ requires Cl, 38.9%).