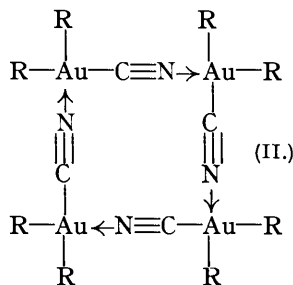
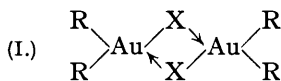


## 22. The Organic Compounds of Gold. Part VIII. Dialkyl Gold Derivatives of Dibasic Acids.

By CHARLES S. GIBSON and WILLIAM T. WELLER.

*Tetraethylsulphatodigold* contains four gold atoms in the molecule. It is soluble in water and in organic solvents. By interaction in aqueous solution with soluble salts of various acids other compounds are obtained and those with typical dibasic aliphatic and aromatic acids are described. The derivatives of oxalic and phthalic acids contain two gold atoms, those of higher members of the  $C_nH_{2n}(CO_2H)_2$  ( $n = 1-6$ ) series four gold atoms, and those of *isophthalic* acid and the corresponding 4-nitro-compound six gold atoms in the respective molecules. Suggestions are made concerning the constitution of those compounds.

THE parent substances of the organic compounds of gold so far studied, although they are non-electrolytes, may be regarded as derivatives of the monobasic hydrochloric, hydrobromic, hydriodic and hydrocyanic acids (see Part VII, J., 1939, 762, for earlier references). These compounds are of two types, the molecular formulæ of which are planar and represented by (I) and (II), where R = alkyl and X = Cl, Br, I (J., 1937, 1690; *Proc. Roy. Soc.*, 1939, A, 173, 147, 160).

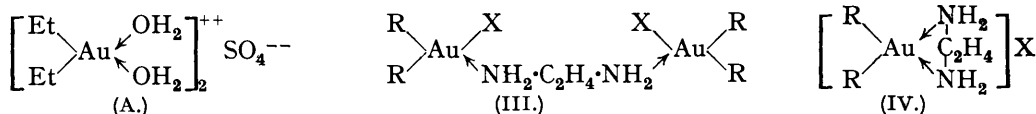


In the course of other work (Gibson, E.P. 497,240) it was shown that, unlike related compounds, the product of interaction of silver sulphate is soluble not only in organic solvents, but also in water. By shaking "di-phenylethyl auric bromide" in benzene with an excess of silver sulphate, Kharasch and Isbell (*J. Amer. Chem. Soc.*, 1931, 53, 2701) obtained "di-phenylethyl auric sulphate," a colourless compound insoluble in water. They determined its gold content and showed that it could be sulphonated. They did not determine the molecular weight, but it was probably a sulphato-compound analogous to that now described.

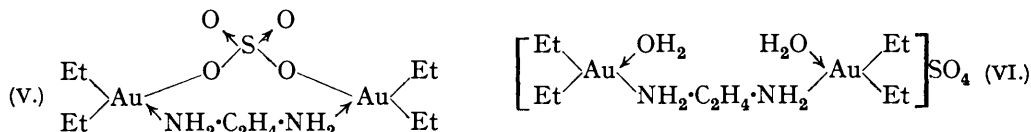
The colourless crystalline compound, readily prepared by shaking an excess of silver sulphate with an acetone solution of diethylmonobromogold, has the expected empirical formula,  $(C_2H_5)_4Au_2SO_4$ . Although it darkens somewhat rapidly on exposure to light, fresh preparations of the compound are easily manipulated. The compound is soluble in such solvents as acetone, alcohol, ethyl acetate and *cyclohexane* and its molecular weight in boiling acetone indicates that the molecular formula is twice that of the empirical formula, so there are four gold atoms in the molecule. The substance may be described as *tetra-*

*ethylsulphatodigold* or more systematically as *bis(tetraethylsulphatodigold)* \*; its constitution is discussed in the following paper. In aqueous solution the compound gives sulphate ions and has a molecular weight in agreement with the constitution (A), in which it is necessary to postulate the co-ordination of two molecules of water in order to preserve the 4-covalency of the auric gold atom. In aqueous solution the compound might be described as diethyldiaquoauric sulphate.

Unlike the dialkylhalogenogold compounds which yield ethylenediamine derivatives of two types—non-electrolytes of the general formula (III) † and electrolytes of the general formula (IV)—the above sulphato-compound yields only one compound with ethylenediamine. This is *ethylenediaminetetraethylsulphatodigold* (V), its molecule containing a



nine-membered heterocyclic ring. It is a colourless compound soluble in bromoform and in water. Since its aqueous solution contains sulphate ions, it is necessary to postulate that in aqueous solution the compound is ionised as indicated (VI).



2 : 2'-*Dipyridyltetraethylsulphatodigold* has a constitution similar to that of the ethylenediamino-compound and, like it, is soluble in water, giving sulphate ions. In this case, as with 2 : 2'-dipyridyltetraethyldibromodigold (Brain and Gibson, *loc. cit.*), the nitrogen atoms are attached to different gold atoms and attempts to prepare compounds in which both nitrogen atoms in dipyriddy are attached to the same gold atom have been unsuccessful.

Attempts to prepare tetramethylsulphatodigold have been unsuccessful and, although the tetra-*n*-butyl compound has been isolated, its instability made analysis and further investigation impossible. *Tetra-n-propylsulphatodigold* has been isolated and its properties are analogous to those of the tetraethyl compound. Its solubility in water appears to be lower than that of the tetraethyl compound and apparently higher than that of the tetra-*n*-butyl derivative. Aqueous solutions of the tetraethyl and the tetra-*n*-propyl compound may also be obtained by treatment of the ethylenediaminodialkylauric bromides with silver sulphate and, after separation of the silver halide, acidification of the filtrate with sufficient sulphuric acid to combine with the ethylenediamine.

Aqueous solutions of the ethyl and the *n*-propyl compound react with aqueous solutions of salts of many acids, *e.g.*, hydrochloroplatinic, hydrochloroauric, acetic, phosphoric and tartaric acids, and also with sodium hydroxide, yielding precipitates; the compounds obtained with the sodium salts of representative dibasic organic acids have been studied in some detail. The colourless precipitates obtained by mixing aqueous solutions of the tetraethyl compound with hydrochloric, hydrobromic and hydriodic acids have been identified as diethylmonochlorogold, diethylmonobromogold, and diethylmonoiodogold respectively.

When aqueous solutions of equivalent quantities of tetraethylsulphatodigold and sodium oxalate are mixed, a colourless precipitate is obtained. This substance crystallises from ligroin in long colourless needles, and analysis and molecular weight determination (freezing cyclohexane) confirm that it is *tetraethyloxalatodigold* (VII) (compare Mann and co-workers,

\* In keeping with the nomenclature adopted in the work on organic compounds of gold, the substances are allotted names based on the simplest, frequently the empirical, formula and not necessarily on the molecular formula.

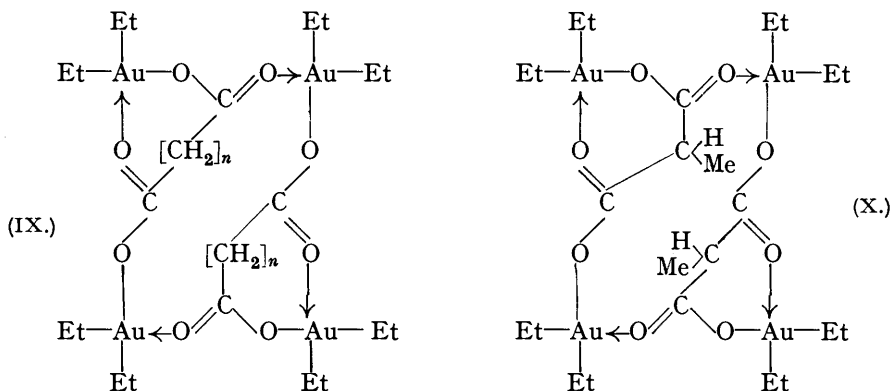
† Previous attempts to prepare *ethylenediaminetetraethyldibromodigold* (as III) have been unsuccessful (compare Brain and Gibson, J., 1939, 762); but this compound has now been obtained. It is colourless and insoluble in water. Like the methyl and *n*-propyl analogues, it decomposes with effervescence on melting.

J., 1938, 2086). This compound is soluble in an aqueous solution of sodium oxalate and when an excess of the latter is used to react with tetraethylsulphatodigold, the precipitate first formed redissolves. The residue obtained by evaporation of the aqueous solution



to dryness at the ordinary temperature, on extraction with boiling ethyl alcohol, yields a sparingly soluble, crystalline compound, presumably the salt, *sodium diethyloxalatodigold* (VIII). The aqueous solution is very stable and undergoes little or no decomposition even on boiling; on acidification with hydrochloric and hydrobromic acid it yields diethylmonochlorogold and diethylmonobromogold respectively. Attempts to prepare an ester corresponding to (VIII), the investigation of which would have afforded further information as to its constitution, have given indeterminate results. Compound (VII) cannot be prepared from diethylmonobromogold and sodium oxalate and this may be due to the fact that, unlike the sulphato-compound, the bromo-compound has little, if any, tendency to become an electrolyte.

The colourless crystalline compound obtained by the interaction of tetraethylsulphatodigold with the calculated quantity of sodium malonate is also a non-electrolyte and has a molecular weight in bromoform which indicates that the compound has four gold atoms in the molecule (compare the constitution of the dialkylcyanogold compounds) and is described as *tetraethylmalonatodigold*. Constitution (IX) ( $n = 1$ ) is provisionally assigned to it. Although the valencies of the gold atoms are planar, the molecule, unlike that of a dialkylmonocyanogold compound, cannot be planar for obvious reasons. This compound is not soluble in an aqueous solution of sodium malonate and it has not been possible to



prepare a malonato-compound analogous to (VIII). Such a malonato-compound would have a ring system somewhat similar to that in the dialkylgoldacetylaceton compounds (J., 1930, 2531; 1939, 762) and the fact that derivatives analogous to (VIII) cannot be derived from compounds containing at least four gold atoms in the molecule may be connected with the stability of such heterocyclic systems, which apparently do not undergo a rearrangement similar to that which takes place when (VII) is converted into (VIII). It has not been possible to study the derivative of monomethylmalonic acid in any detail. The product obtained by mixing aqueous solutions of equivalent quantities of sodium monomethylmalonate and tetraethylsulphatodigold could not be recrystallised and its molecular weight was not determined. Analysis showed that the substance was *tetraethylmonomethylmalonatodigold* and, in all probability, its constitution is that indicated (X).

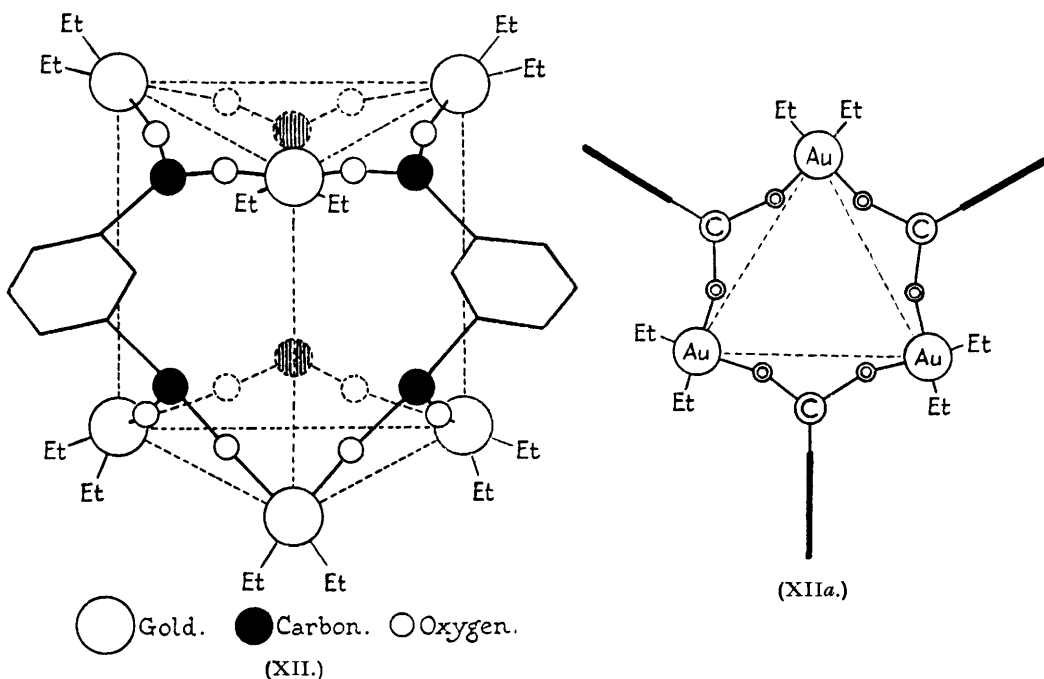
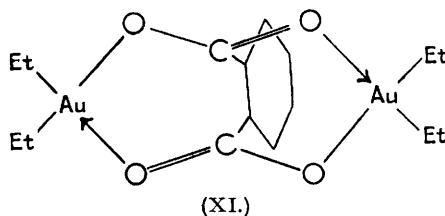
Analogous compounds of higher homologues of the normal acids of the  $C_nH_{2n}(CO_2H)_2$  series ( $n = 2-6$ ) have been investigated in the case of the  $n$ -propyl derivatives. As none of the five compounds is soluble in an aqueous solution of the sodium salt of the particular acid, excess of the latter could be employed in the preparation. The molecular weights of *tetra-n-propylsuccinatodigold*, *tetra-n-propylglutaratodigold*, *tetra-n-propyladipatodigold*,

*tetra-n-propylpimelatodigold* and *tetra-n-propylsuberatodigold* have been determined. In each case there are four gold atoms in the molecule, which may be represented conventionally by (IX) (Pr<sup>a</sup> instead of Et,  $n = 2-6$ ). No attempt is made at present to indicate the disposition of the  $-[\text{CH}_2]_n-$  chains relative to the rest of the molecule. The last two compounds are particularly stable with definite melting points of the same order as those of pimelic and suberic acids themselves.

*Tetraethylsaccharatodigold* has been prepared; its solubility in water (about 0.2%) is surprisingly small. This compound proved to be too unstable in bromoform to permit a determination of its molecular weight.

Since attempts to isolate an azelato-derivative ( $n = 7$ ) have been unsuccessful, it may be that the derivatives of acids higher in the series than suberic acid cannot be prepared.

Representative derivatives of aromatic dicarboxylic acids have also been studied for comparison with those of the aliphatic acids. *Tetraethylphthalatodigold*, prepared from tetraethylsulphatodigold and an excess of sodium phthalate in aqueous solution, has a molecular weight in ethylene bromide indicating the presence of two gold atoms in the molecule and, to this extent, this compound is analogous to the oxalato-derivative (VII). It is, however, insoluble in an aqueous solution of sodium phthalate and is particularly stable. Its constitution is represented schematically by (XI); a rough calculation indicates that the  $\text{O} \angle \text{Au}$  angle is probably not widely different from  $90^\circ$  and it is also



probable that the two gold atoms are in the same plane. *Tetraethyl-3-nitrophthalatodigold* proved to be too sparingly soluble for the determination of its molecular weight.

*Tetraethylisophthalatodigold*, prepared in the usual manner, is a colourless crystalline non-electrolyte insoluble in an aqueous solution of sodium *isophthalate*; it is more soluble in organic solvents than the phthalato-compound. Its molecular weight in freezing *cyclohexane* indicates that the molecule contains six gold atoms and this is also true of the molecule of *tetraethyl-4-nitroisophthalatodigold*. Substances containing six gold atoms in the molecule have not been previously described. Of various constitutions which can be suggested for such molecules, the most likely would appear to be one in which the gold atoms are at the corners of a regular triangular prism represented by (XII) and (XIIa), each *isophthalato*-group being placed as shown. In (XII) two of the *isophthalato*-residues are shown in full and the carboxyl groups of the third (behind the back face of the prism) by broken circles. (XIIa) is a projection of the same structure on the triangular base of the model.

To complete this series *tetraethylterephthalatodigold* has been prepared, but owing to its very low solubility no information regarding its molecular weight is available.

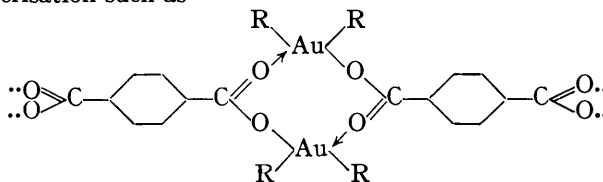
The co-ordinate link which each  $\begin{matrix} R \\ R \end{matrix} \text{Au-}$  group requires for stability in the compounds

now described is not obtained by the formation of four-membered rings,  $\begin{matrix} R \\ R \end{matrix} \text{Au} \begin{matrix} \diagup \text{O} \\ \diagdown \text{O} \end{matrix} \text{C-}$ .

Otherwise, it would be expected that each of the derivatives of the dicarboxylic acids would have a molecular weight indicating the presence of two gold atoms in the molecule and the three phthalic acid compounds would all have the same molecular weight. The co-ordinate link must therefore come from a second carboxyl. When the carboxyl groups are adjacent as in oxalic and phthalic acids, the molecule contains two gold atoms. In the case of the higher aliphatic acids the distance between the two carboxyl groups necessitates polymerisation from two to four gold atoms in the molecule. In the case of the *isophthalic acid* (and of the corresponding 4-nitro-compound) derivative polymerisation to six gold atoms in the molecule becomes necessary. The polymerisation in this case is greater than in the case of the derivative of glutaric acid, which has, however, a carbon

skeleton,  $-\text{O}_2\text{C}-\text{CH}_2-\text{CH}_2-\text{CO}_2-$ , similar to that in *isophthalic acid*,  $-\text{O}_2\text{C}-\text{CH}=\text{CH}-\text{CO}_2-$ .

The very low solubility of the terephthalic acid derivative could be associated with unlimited polymerisation such as



#### EXPERIMENTAL.

*Tetraethylsulphatodigold*.—Diethylmonobromogold (3 g.), dissolved in acetone (50 ml.), and powdered silver sulphate (3.5 g., three times the calculated quantity) were shaken until the reaction was complete (absence of bromo-compound in the solution; 4–5 hours). The filtered solution was evaporated in a dry atmosphere under reduced pressure at the ordinary temperature, and the residue recrystallised from the minimum quantity of ethyl acetate, the product (average yield, 2.0 g.; 73%) being obtained in small colourless needles, m. p. 96–97° (decomp.) (Found : C, 16.2; H, 3.7; S, 5.9; Au, 64.6.  $\text{C}_8\text{H}_{20}\text{O}_4\text{SAu}_2$  requires C, 15.8; H, 3.3; S, 5.3; Au, 65.0%).

*Tetraethylsulphatodigold* is soluble in water, acetone and alcohol, sparingly soluble in benzene and *cyclohexane*, and almost insoluble in ligroin [Found, cryoscopic in water : *M*, 230. Calc. for the compound fully dissociated into three ions as indicated above : *M*, 226. Found, ebullioscopic in acetone by the Landsberger-Walker method (*c*, 12.77, 12.7) : *M*, 1235, 1360. Calc. for  $\text{C}_{16}\text{H}_{40}\text{O}_8\text{S}_2\text{Au}_4$  : *M*, 1212].

*Ethylenediaminetetraethylsulphatodigold* (V) was prepared by mixing an acetone solution of the sulphato-compound with an alcoholic solution of ethylenediamine (2 mols.). The colourless precipitate was washed with acetone. It may be recrystallised from ethyl alcohol (Found :

C, 18.35; H, 4.3; N, 4.4; S, 5.4; Au, 58.4.  $C_{10}H_{28}O_4N_2SAu_2$  requires C, 18.0; H, 4.2; N, 4.2; S, 4.8; Au, 59.2%). The compound decomposes violently at about 147°. It is soluble in water and bromoform and almost insoluble in acetone and benzene.

The corresponding bromo-compound, *ethylenediaminotetraethyltribromodigold* (footnote, p. 103), was prepared by adding a ligroin solution of diethylmonobromogold (1 g.) to ethylenediaminodiethylgold bromide (1.18 g.) dissolved in methanol. The clear solution was evaporated under reduced pressure, and the residue extracted with light petroleum (b. p. 40–60°) to remove any unchanged diethylmonobromogold. The solid product was dissolved in purified acetone, and the filtered solution evaporated under reduced pressure in a dry atmosphere (Found: Br, 21.5; Au, 53.6.  $C_{10}H_{28}N_2Br_2Au_2$  requires Br, 21.9; Au, 54.0%).

It was also prepared by mixing ethylenediamine (1 mol.) in ethyl alcohol with diethylmonobromogold (1 g.) in ligroin (15 ml., b. p. 60–80°). The colourless precipitate was washed with ligroin and dissolved in acetone, and the solution evaporated as described above (Found: Au, 53.2%).

The colourless compound is insoluble in water, sparingly soluble in ligroin and chloroform and soluble in alcohol and acetone. It decomposes with effervescence at about 113–114°.

2:2'-Dipyridyltetraethylsulphatodigold (as V) was prepared in good yield by mixing ethyl-alcoholic solutions of the sulphato-compound (0.5 g.) and 2:2'-dipyridyl (0.13 g.; 2 mols.) and evaporating the solution to dryness at the ordinary temperature. Excess of dipyridyl was removed by extraction with ligroin, the residue dissolved in the minimum quantity of ethyl alcohol, and acetone added to the filtered solution until, after cooling, colourless needles (0.4 g.), m. p. 162–163° (decomp.), separated (Found: C, 28.1; H, 3.6; N, 3.9; Au, 51.6.  $C_{18}H_{28}O_4N_2SAu_2$  requires C, 28.3; H, 3.7; N, 3.7; Au, 51.7%).

The compound is soluble in water, alcohol and acetone; it is less soluble in *cyclohexane* and apparently insoluble in ethyl acetate, ligroin and chloroform.

*Tetra-n-propylsulphatodigold*.—The solution obtained by shaking silver sulphate (16 g.) with di-*n*-propylmonobromogold (16 g.) in acetone (100 ml.) for 4½ hours was evaporated under reduced pressure over sulphuric acid at the ordinary temperature. The somewhat gummy residue, soluble in all the usual organic solvents, was extracted with warm light petroleum (b. p. 40–60°), and this filtered solution evaporated under reduced pressure in a desiccator. The residue was dissolved in a small quantity of methanol, and water added to the filtered solution until crystallisation began (Found: Au, 59.9.  $C_{12}H_{28}O_4SAu_2$  requires Au, 59.5%).

This compound is less soluble in water and less stable than the ethyl compound; it is more soluble in organic solvents and can be recrystallised from *cyclohexane*, in which, however, it is too sparingly soluble at the freezing point for molecular weight determination.

Following the usual procedure and taking advantage of its apparently low solubility in water and high solubility in organic solvents, attempts were made to isolate tetra-*n*-butylsulphatodigold, but the compound was not obtained in a form suitable for analysis. The attempted preparation of the tetramethyl compound starting with dimethylmoniodogold was unsuccessful.

*Tetraethyloxalatodigold* (VII) was obtained as a colourless precipitate (0.43 g.) on mixing aqueous solutions of tetraethylsulphatodigold (0.5 g.) and of an equivalent quantity of sodium oxalate (0.113 g.). It was recrystallised from light petroleum (b. p. 40–60°) and obtained in long colourless needles, m. p. 81° (explosive decomp. at about 120°). The molecular weight was determined in freezing *cyclohexane* (*c* 2.194) (Found: Au, 65.9; *M*, 568.  $C_{10}H_{20}O_4Au_2$  requires Au, 65.9%; *M*, 598).

Tetraethyloxalatodigold is insoluble in water and soluble in the usual organic solvents. It undergoes decomposition even in the dark.

*Sodium Diethyloxalatogold* (VIII).—The sulphato-compound (0.7 g.) in water (10 ml.) was added to an aqueous solution of sodium oxalate (0.78 g.; 5 mols.) containing a little acetone to facilitate solution of (VII) which is first precipitated. The residue from evaporation of the clear solution to dryness at the ordinary temperature was extracted several times with boiling ethanol, and this solution filtered and evaporated (crude yield, 0.45 g.). The compound was recrystallised from ethanol and obtained in colourless soft needles (Found: Na, 6.0; Au, 53.4.  $C_8H_{10}O_4NaAu$  requires Na, 6.3; Au, 53.9%).

The remaining compounds were all prepared by mixing aqueous solutions of the particular sulphato-compound and of the dibasic acid exactly neutralised with sodium (or potassium) hydroxide. Unlike (VII), these compounds are all insoluble in an excess of an aqueous solution of the salt of the dibasic acid, and an excess of the latter was used in many cases. When the compound could be crystallised, the solvent used is given in parentheses; otherwise, water-soluble substances were removed by thorough washing with water. The yields were practically

quantitative and all the substances are insoluble or, in the case of the saccharato-compound, only slightly soluble, in water.

*Tetraethylmalonatodigold* (as IX,  $n = 1$ ) crystallises in colourless needles (acetone) which explode without melting at about  $136^\circ$ . It is soluble in bromoform and sparingly soluble in cyclohexane [Found: C, 21.3; H, 3.5; Au, 64.4;  $M$  in freezing bromoform ( $c$  1.197), 1150.  $C_{22}H_{44}O_8Au_4$  requires C, 21.5; H, 3.5; Au, 64.3%;  $M$ , 1226].

*Tetraethylmonomethylmalonatodigold* (X) was obtained as a colourless precipitate and not recrystallised. Its melting was accompanied by decomposition ( $90-100^\circ$ ) and it decomposed explosively at about  $140^\circ$ . It was sparingly soluble in acetone and readily soluble in chloroform. Its molecular weight was not determined [Found: C, 23.3; H, 4.1; Au, 62.3.  $C_{12}H_{24}O_4Au_2$  requires C, 23.0; H, 3.8; Au, 62.9%].

*Tetra-n-propylsuccinatodigold* (as IX,  $n = 2$ ), obtained as a colourless precipitate, has m. p.  $145-146^\circ$  (decomp.). It is soluble in ligroin, cyclohexane and benzene. It is unstable in bromoform [Found: C, 27.8; H, 4.4; Au, 57.6;  $M$  in freezing cyclohexane ( $c$  1.116) and benzene ( $c$  3.955), 1387, 1280.  $C_{32}H_{64}O_8Au_4$  requires C, 28.1; H, 4.7; Au, 57.8%;  $M$ , 1366].

*Tetra-n-propylglutaratodigold* (as IX,  $n = 3$ ) crystallises in slightly discoloured needles (acetone). Its melting is accompanied by decomposition (about  $90^\circ$  onwards) [Found: C, 29.4; H, 4.9; Au, 56.2;  $M$  in freezing cyclohexane ( $c$  1.617), 1307.  $C_{34}H_{68}O_8Au_4$  requires C, 29.3; H, 4.9; Au, 56.6%;  $M$ , 1394].

*Tetra-n-propyladipatodigold* (as IX,  $n = 4$ ) crystallises in colourless needles (acetone), m. p. indef. ( $124-132^\circ$ , decomp.). It is soluble in cyclohexane and benzene [Found: C, 29.7; H, 4.7; Au, 55.4;  $M$  in freezing cyclohexane and benzene, 1311 ( $c$  1.621), 1243 ( $c$  1.372), 1282 ( $c$  3.769).  $C_{36}H_{72}O_8Au_4$  requires C, 30.4; H, 5.1; Au, 55.5%;  $M$ , 1422].

*Tetra-n-propylpimelatodigold* (as IX,  $n = 5$ ) crystallises in colourless soft needles (acetone), m. p.  $79-81^\circ$  (decomp. at about  $100^\circ$ ) [Found: C, 32.1; H, 5.5; Au, 54.2;  $M$  in freezing cyclohexane, 1295 ( $c$  1.846).  $C_{38}H_{76}O_8Au_4$  requires C, 31.5; H, 5.3; Au, 54.4%;  $M$ , 1450].

*Tetra-n-propylsuberatodigold* (as IX,  $n = 6$ ) crystallises in colourless needles (acetone), m. p.  $86-88^\circ$  (decomp. from about  $110^\circ$ ) [Found: C, 32.6; H, 5.4; Au, 53.4;  $M$  in freezing cyclohexane, 1341 ( $c$  1.546).  $C_{40}H_{80}O_8Au_4$  requires C, 32.5; H, 5.5; Au, 53.4%;  $M$ , 1478].

*Tetraethylsaccharatodigold* crystallises in colourless needles (acetone-ethanol) and is not stable. It is sparingly soluble in cyclohexane, readily soluble in chloroform and unstable in bromoform; its molecular weight could not be determined (Found: C, 23.4; H, 3.9; Au, 55.3.  $C_{14}H_{28}O_8Au_2$  requires C, 23.4; H, 3.9; Au, 54.9%).

*Tetraethylphthalatodigold* (XI) was obtained as a colourless precipitate. It decomposes without melting from about  $120^\circ$  but at the ordinary temperature is very stable. It is sparingly soluble in the ordinary organic solvents, soluble in ethylene bromide and also in acetic acid, by which it is decomposed [Found: C, 28.9; H, 4.0; Au, 58.3;  $M$  in freezing ethylene bromide ( $c$  0.4543, 0.4125), 577, 640.  $C_{16}H_{24}O_4Au_2$  requires C, 28.5; H, 3.6; Au, 58.5%;  $M$ , 674].

*Tetraethyl-3-nitrophthalatodigold*, obtained as a colourless precipitate, was very sparingly soluble in organic solvents and its molecular weight could not be determined (Found: Au, 54.5.  $C_{16}H_{23}O_6NAu_2$  requires Au, 54.9%).

*Tetraethylisophthalatodigold* (XII and XIIa), obtained as a colourless precipitate, is sparingly soluble in acetone and ethanol and readily soluble in chloroform, bromoform and cyclohexane. It crystallises in colourless needles (chloroform-acetone) and on heating decomposes without melting [Found: Au, 57.9;  $M$  in freezing cyclohexane, 1793 ( $c$  1.206), 1875 ( $c$  2.228).  $C_{46}H_{72}O_{12}Au_6$  requires Au, 58.5%;  $M$ , 2022].

*Tetraethyl-4-nitroisophthalatodigold* (as XII and XIIa), obtained as a colourless precipitate sparingly soluble in the usual organic solvents, was sufficiently soluble in freezing cyclohexane for molecular weight determination [Found: Au, 54.5;  $M$ , 2210 ( $c$  0.9890).  $C_{48}H_{69}O_{18}N_3Au_6$  requires Au, 54.9;  $M$ , 2187].

*Tetraethylterephthalatodigold*, obtained as a colourless precipitate, decomposes without melting and is very sparingly soluble in the usual organic solvents. Its molecular weight could not be determined (Found: Au, 58.4.  $C_{16}H_{24}O_4Au_2$  requires Au, 58.5%).

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