

pH measurements 50 c.c. of one per cent. solution of sodium chloride was shaken with 0.5 gram of the earths. The table given above gives some of the results which are graphically shown in Fig. 1.

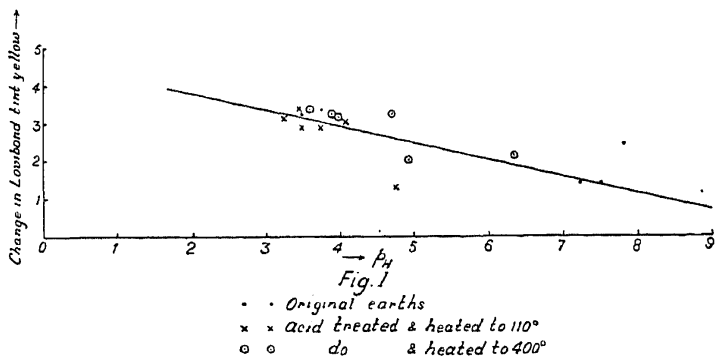


Fig. 1.

The accuracy of the colour measurements as compared with the accuracy of the pH measurements by the quinhydrone electrode is limited by the use of the Lovibond Tintometer and accounts for the discrepancies. Our results, however, indicate that the decolourising action and the activation by acids of these earths are directly related with the exchangeable hydrogen contained in the earth. While thus indirectly supporting the conclusion of Fogle and Ohn regarding the zeolitic nature of the active ingredient in the fuller's earth, our results show that it is not "calcium" zeolite but "hydrogen" zeolite that is active in decolourising. It is possible that the action of the zeolite is replaced by the hydrogen ion during the process of activation by acids, the hydrogen in turn being replaced by the sodium ion when treated with the salt solution, both reactions being governed by the law of mass action. The "hydrolytic adsorption," by other clays can be explained on the same basis. Further work is in progress.

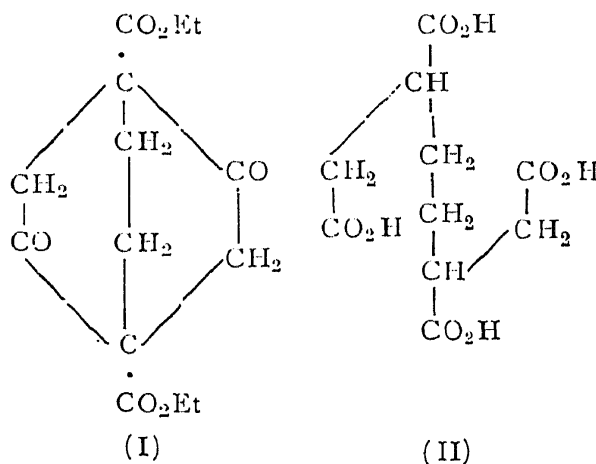
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p-Bridging of Succinosuccinic Ester.

CONSIDERING the products obtainable from succinosuccinic ester by bridging the *p*-carbon atoms with methylene iodide or ethylene bromide "to be of extremely great interest," Baeyer¹ tried the experiments evidently without success. The non-formation of a strainless tricyclic system from bicyclic nonanedionetetracarboxylic ester, alike Baeyer's failure, were attributed by Meerwein² to the splitting up of the bridged compounds by alkali.

After a large number of attempts made under varying experimental conditions, the desired bridge formation has now been effected by boiling dry sodio-derivative of succinosuccinic ester under reflux during 72 hours with ethylene and trimethylene bromides, the resulting compounds melting at 112° and 132° respectively. That the reactions have not taken place in the enolic phase, is proved by the fact that the bridged compounds give sharp melting disemicarbazones. The bridged esters are easily hydrolysed to the corresponding diacids, m.p. 274° and 238° respectively by boiling with dilute HCl (1:1) and these acids, in turn, give sharp melting disemicarbazones. It is interesting to note that the bridged esters cannot be decarboxylated under conditions in which succinosuccinic ester readily gives 1:4-diketohexamethylene. The compound, m.p. 112° (I) on treatment with 10 per cent. alcoholic potash suffers ring fission to yield, what appears from combustion analysis and equivalent determination to be $\beta\beta'$ -dicarboxysuberic acid, m.p. 170° (II) as yet unknown and on treatment with 1.5 per cent.



¹ Welds, *Z. Angew. Chem.*, 1927, **40**, 7982.
² Haseman, *J. Phys. Chem.*, 1929, **33**, 1514.
³ Neuman, *Z. Angew. Chem.*, 1927, **40**, 337.
⁴ Thurman, *Ind. Eng. Chem.*, 1932, **24**, 1189.
⁵ Fogle and Ohn, *Ind. Eng. Chem.*, 1933, **25**, 1070.
⁶ Bancroft, *Applied Colloid Chemistry*, 3rd Ed., p. 146, McGraw-Hill, 1932.
 Also Cf. Chameron, *J. Phys. Chem.*, 1910, **14**, 400.
⁷ Burgharat, *Ind. Eng. Chem.*, 1931, **23**, 801.

neutral permanganate it gives an acid, m.p. 150° which, however, seems to be

different from adipic acid. Further work is being continued.

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¹ Baeyer, *Ber.*, 1892, 25, 2123.

² Meerwein, *J. Pr. Chem.*, 1922, (ii), 104, 180.

A Synthesis of *Cis*- and *Trans*-*dl*-1-*iso*-Propyl Cyclopropane-1:2-Dicarboxylic Acids and a Resolution of the *Cis* Acid. Synthesis of Umbellularic Acid.

In his experiments on the constitution of umbellulone, Tutin¹ obtained as the ultimate product of its oxidation, an optically active dibasic acid, umbellularic acid, $C_8H_{12}O_4$, m.p. 120–121° (α_D) – 89.7° (in $CHCl_3$). Although Tutin believed the acid to be a derivative of methyl *cyclo*-pentane, the experiments of Semmler² definitely fixed its constitution as 1-*isopropyl cyclopropane*-1:2-dicarboxylic acid. The *cis*- and *trans*-*dl*-forms of this acid have now been synthesised.* Also the *cis* acid has been resolved into its optical antipodes and the properties of the latter are identical with those of umbellularic acid.¹

Ethyl α -*isopropyl acrylate*³ adds on ethyl diazoacetate to give ethyl 5-*isopropyl*- Δ pyrazoline-3:5-dicarboxylate, b.p. 158° at 1 mm. and the latter splits off nitrogen at 200°⁴ giving rise to the mixed ester, b.p. 144–48°/28 mm. On hydrolysis this furnishes *trans*-*dl*-1-*isopropyl cyclopropane*-1:2-dicarboxylic acid (m.p. 195°·0 C.)[†] in about 35% yield, the rest being a liquid mixture of unsaturated acids. The *cis* anhydride prepared from *trans* acid by heating with acetylchloride at 180°, boils at about 140°/20 mm. The *cis*-*dl*-acid crystallises from water with one molecule of the solvent (m.p. 95°, sintering 86°). The anhydrous acid melts at 124–125° and passes over to the anhydride at 150°. The chemical properties of these two acids are identical with those of umbellularic acid.¹

On combining *cis*-*dl*-acid with brucine in aqueous solution the salt of the *d*-form separated first, and had the composition $C_{54}H_{64}O_{12}N_4 \cdot 9H_2O$ (α_D^{30}) – 25.63 (in alcohol). The active acid liberated from the salt had (α_D^{31}) + 87.7 (in $CHCl_3$), m.p. 118° with $1H_2O$ about 83°. For obtaining the *l*-acid cinchonidine was employed, when the salt of the *l* form $C_{27}H_{34}N_2O_5$, separated first. The acid liberated had (α_D^{31}) – 81°·13.

The crystalline forms of both the antipodes were in agreement with those cited by Tutin for umbellularic acid.

Full details of the work have been sent for publication elsewhere.

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¹ Tutin, *J.C.S.*, 1906, 89, 1104.

² Semmler, *Ber.*, 1907, 40, 5019; *Ibid.*, 1908, 41, 3988.

³ Blaise and Luttringer, *Bull. Soc. Chim.*, 1905, (3), 33, 648, 776.

⁴ Buchner and Papendieck, *Annalen*, 1893, 273, 232. von Auwers and König, *Ibid.*, 1932, 496, 252.

* The Synthesis of the *dl*-acids was complete and the resolution of the *cis* acid was in hand when an abstract on the synthesis of umbellularic acid appeared (Rydon, *Chem. and Ind.*, 1936, 55, 294). The method adopted by Rydon, however, is different and we thought it fit to continue our work and place the results for publication.

† The figures for melting point given in this note are all uncorrected.

Apparatus for the Measurement of Respiratory Exchange in Plants.

FOR the measurement of respiratory gaseous exchange in plants Haldane's gas-analysis apparatus is commonly employed. Although highly accurate, the apparatus in its original form¹ is inconvenient and rather cumbersome for respiration studies in plants. Carpenter² has replaced the long, cylindrical levelling tube which is rather difficult to manipulate, by a small mercury bulb which is easily handled. The original Haldane apparatus contains a combustion pipette for the oxidation of carbon monoxide or methane. This pipette was utilised to advantage by Carpenter² for the absorption of oxygen by means of moist phosphorus. It has the advantage over potassium pyrogallate that it does not have to be renewed so frequently and that the absorption can be carried out without the continuous raising and lowering of the mercury levelling bulb. In this Laboratory, however, during the course of investigations on the gas-storage of tropical fruits the apparatus has been further simplified and the technique for the measurement of respiratory exchange in plants considerably improved. The gas sampler employed by Haldane has been dispensed with, the measuring pipette serving as a sampling appliance as well. The potassium pyrogallate bulb (together with its accessory bulbs) has been replaced by a phosphorus bulb of the type employed by Carpenter,²