NOTES.

Preparation of Hexadecanesulphonic Acid. By ROGER C. MURRAY.

THE following method for the prep. of hexadecanesulphonic acid from the mercaptan gives a much more nearly quant. yield with less trouble than the published processes (Reychler, *Bull. Soc. chim. Belg.*, 1913, **27**, 110; Norris, J., 1922, **121**, 2161).

Hexadecyl iodide was added in small quantities with const. shaking to a solution of KSH (better than NaSH) in EtOH on the water-bath. The greyish-white product, cryst. from EtOH–EtOAc, gave colourless needles, m. p. 47—48°, consisting of a mixture of hexadecyl mercaptan (*ca.* 75%) and dihexadecyl disulphide, owing to the ready oxidation of the former to the latter (private communication from Prof. Hilditch).

With $Pb(OAc)_2$ in EtOH, it gave a yellow ppt. of lead hexadecylmercaptide (Found: Pb, 28·1. Calc., 28·7%). The pure disulphide gives no ppt., which probably explains Fridau's statement (*Annalen*, 1852, **83**, 1), his substance being entirely dihexadecyl disulphide.

Hexadecanesulphonic acid was prepared by the oxidation, which is vigorous, of the mercaptan–disulphide mixture or the pure disulphide with fuming HNO_3. The resulting solution was evaporated to small vol., and the acid finally dried to const. wt. over CaO in a high vac. It crystallised well from 50% aq. EtOH. The solid acid contained 5% of H_2O, was slightly brown, and gave clear aq. solutions, which curded on cooling. Unless the reaction mixture was heated for some time at 100°, the aq. solution of the sulphonic acid or its sodium salt was opalescent, possibly owing to the presence of not fully oxidised material, probably dihexadecyl disulphoxide. In the case of the sodium salt the cloudiness could be removed only by evaporation to dryness with aqua regia, six crystns. from EtOH being ineffective. The sulphonic acid (yield, *ca.* 98%) is very much more sol. than the sodium salt, in common with other org. sulphonic acids, and its gelling, or curding, power is very much greater and less sensitive to impurities. This is presumably due to a difference in the hydration of the H and Na ions.

The hexadecanesulphonate radical can be estimated as the barium salt, the solubility of which, determined by washing the salt with boiling H_2O, is about 4 times that of $BaSO_4$. The ppt. must be kept at least 8 hr. before filtration and then dried at 120°.

The conductimetric titration curve of the acid is very similar to that of HCl.

The statement of Reychler (*loc. cit.*), that the reaction between hexadecyl mercaptan and HNO_3 is explosive, has not been confirmed.

The author wishes to acknowledge the helpful suggestions and constructive criticism accorded him by Mr. G. S. Hartley, M.Sc.—THE SIR WILLIAM RAMSAY LABORATORIES OF PHYSICAL AND INORGANIC CHEMISTRY, UNIVERSITY COLLEGE, LONDON. [*Received, April 13th,* 1933.]

The So-called 1 : 2-*Dihydropapaverine.* By JOHANNES S. BUCK.

THE interpretation given by Young and Robinson (this vol., p. 275) of the reactions involved in the synthesis of papaverine from homoveratroyl-ω-aminoacetoveratrone (Buck, *J. Amer. Chem. Soc.*, 1930, **52**, 3610) is undoubtedly correct. The author has carried out mixed m. p. determinations with his " 6 : 7 : 3′ : 4′-tetramethoxy-4-hydroxy-1 : 2 : 3 : 4-tetrahydroprotopapaverine " and authentic homoveratro-β-veratrylethylamide, and also with his " 6 : 7 : 3′ : 4′-tetramethoxy-9-keto-1 : 2-dihydroprotopapaverine " and authentic 6 : 7 : 3′ : 4′-tetramethoxy-1-benzoyl-3 : 4-dihydro*iso*quinoline (J., 1924, **125**, 2184). In neither case was there a depression, and the pairs were otherwise identical. These observations confirm the views of Young and Robinson.—TUCKAHOE, NEW YORK, U.S.A. [*Received, May 18th,* 1933.]

Isolation of Crystalline Atisine. By J. P. C. CHANDRASENA.

TUBERS of *Aconitum heterophyllum* grown in Northern India were treated by Jowett's method (J., 1896, **69**, 1518) and the resinous mass obtained was dried in vac., or the moist substance spread on porous plate. A solution of the resulting powder in EtOH was boiled under reflux (charcoal), concentrated, treated with EtOAc until cloudy, warmed till clear, and allowed to cool. Clusters of small needles separated, m. p. 314° (decomp.), $[\alpha]^{29°}_{5461}$ + 16·6° in 90% EtOH [Found : C, 69·2; H, 8·9; N, 3·7. $C_{22}H_{35}O_4N$ requires C, 70·0; H, 9·3; N, 3·7%. $C_{22}H_{31}O_2N$ (Adler's formula) requires C, 77·4; H, 9·1; N, 4·1%]. The substance has no methoxy-groups, and suffers no loss in wt. when heated for 2 days over P_2O_5 at 78°.

Atisine Perchlorate.—The crude alkaloid was dissolved in dil. HCl and warmed with charcoal on the water-bath, and $NaClO_4$ aq. added to the filtered solution. The white ppt. obtained was thrice crystallised from hot H_2O and formed light, white, silky feathers, m. p. 199° (Found : Cl, 8·0. $C_{22}H_{31}O_2N,HClO_4$ requires Cl, 8·0%. $C_{22}H_{35}O_4N,HClO_4$ requires Cl, 7·3%).

The study of atisine is being continued.—UNIVERSITY COLLEGE, COLOMBO, CEYLON. [*Received, April 27th,* 1933.]