

15 ml of water 0.15 g of 10 % Pd on carbon catalyst was added and the reaction mixture was hydrogenated at room temperature until the hydrogen uptake became negligible. The catalyst was removed by filtration, and VI precipitated by addition of acetic acid to the filtrate to pH 6.5. Yield: 0.8 g; m.p. 248.5°. Several recrystallization from aqueous ethanol raised the m.p. to 250°. NMR spectrum, *cf.* Table 1. (Found: C 39.81; H 5.20; N 14.06. Calc. for $C_{13}H_{20}N_4O_6S_2$ (392.44): C 39.79; H 5.13; N 14.28).

1. Lespagnol, A., Bar, D. and Mizon-Capron, Ch. *Pharm. Acta Helv.* **38** (1963) 561.
2. Chattaway, F. D. *J. Chem. Soc.* **95** (1909) 1065.
3. Lespagnol, A., Bar, D., Lespagnol, Ch. and Anouvilh, A. *Bull. Soc. Chim. France* **1965** 800.
4. Oliver, S. C. *J. Rec. Trav. Chim.* **37** (1918) 307.
5. Lespagnol, A., Lespagnol, Ch., Willecomme, B. and Roche J. *Compt. Rend.* **265** (1967) 755.

Received July 29, 1968.

Preparation of Average Sample Solution of Heterogeneous Organic Materials for Determination of Microquantities of Mercury Using Purified Sodium Hydroxide

V. LIDUMS and U. ULFVARSON

Statens Provningsanstalt, Stockholm, Sweden

Mercury is often very unevenly distributed in organic materials. Small samples taken out from different parts of, *e.g.* a fish body do not give exactly the same mercury value. Other, more heterogeneous materials require a number of analyses to obtain the right average value.

A fast and simple way to prepare a representative average sample for mercury determination with the direct combustion-photometric method developed by Lidums and Ulfvarson¹ is to heat the material with sodium hydroxide. Since commercial sodium hydroxide (analytical grade) con-

tains relatively large amounts of mercury, it is impossible to use it for this purpose. A sodium hydroxide solution giving low enough mercury blanks is prepared as follows.

Sodium carbonate or preferably sodium hydrogen carbonate is heated in an electric furnace at 700°C for 1–2 days. Calcium carbonate is heated at 1000–1100°C for about 5 h. 170 g of the obtained calcium oxide are transferred to a 2 liter flask and 60 ml of water are added carefully in small portions (double-distilled water is used exclusively). Then 260 g of the sodium carbonate and 800 ml of water are added. The contents of the flask are heated to boiling and the flask is swirled from time to time until all the sodium carbonate is dissolved. After rapid cooling the mixture is transferred to a polyethylene bottle and calcium carbonate is allowed to settle. The clear solution, containing 20–25 % sodium hydroxide, is then decanted.

For the average sample solution a desired amount of the material is weighed and transferred to a Kjeldahl flask of suitable size. The sodium hydroxide solution is added taking 5 ml for each gram of the sample material. The flask is then heated gently until a homogeneous solution is obtained (for fish it takes only a few minutes). The solution is transferred to a weighed polyethylene bottle with as little water as possible. The total amount of the final solution is determined by weighing. A suitable part of the average sample is weighed in a porcelain boat and analysed according to the procedure described by Lidums and Ulfvarson.¹ The mercury blank of the reagents and vessels is determined by carrying out all the operation steps with the same quantity of the sodium hydroxide solution as used for preparation of the average sample.

The method has been tested using a fish, previously analysed by various laboratories. The average mercury content, calculated from 39 single determinations, was found to be 841 ng Hg per gram. A sample solution was prepared taking specimens from different parts of this fish — together 4 g. Three single samples were taken out from the average solution and the analysis of them gave the following values: 830–815–830 ng Hg per g.

1. Lidums, V. and Ulfvarson, U. *Acta Chem. Scand.* **22** (1968) 2150.

Received August 15, 1968.