mixture is shaken and heated gently to drive out water and then to a dull red (above 452°, the m. p. of tellurium). The mixture is held at this high temperature for about five minutes with occasional shaking during the progress of heating. The flask is allowed to cool somewhat and then plunged into 100 ml. of water in a large mortar. The flask breaks and both it and the solid contents are coarsely powdered with a pestle. The aqueous layer with suspended tellurium is transferred to a liter erlenmeyer flask with suction and the residue in the mortar extracted with several 50-ml. portions of hot water until only minor amounts of radioactive materials are being transferred. The extracts and washings are heated to boiling to coagulate the precipitate. The solution is cooled and filtered to remove tellurium.

The remainder of the isolation requires an all-glass-standard taper distilling apparatus consisting of a 500-ml. flask, an adapter with addition tube, an adapter and condenser and a receiving adapter arranged to dip into a receiving solution. The filtered alkaline extract is run into the distilling flask, which contains a few boiling stones, and evaporated to a volume of about 50 ml. The distillate contains no radioactive material and is discarded. A concentrated solution of potassium permanganate is added in excess to oxidize all iodine in the alkaline solution to iodate. The receiving flask is changed to one containing a dilute solution of sodium sulfite and sodium carbonate (about 1 mg. of each in several ml. of water). The delivery tip is covered by this solution. Fifty ml. of concentrated sulfuric acid is added through the addition tube. Care is necessary because of the heat developed. After the sulfuric acid has been added, 1 g. of oxalic acid dissolved in a minimal amount of hot water is added. The iodate is reduced to iodine. The mixture is now distilled until substantially all of the radioactivity has collected in the receiver where the iodine is converted to iodide. The solution of iodide containing some carbonate, sulfite and sulfate is used directly in most of our work.

The tellurium residue when dissolved in nitric acid containing a small amount of chloride ion, gave, in the presence of excess dissolving silver, a silver chloride precipitate which contained relatively little radioactivity. When unirradiated tellurium to which I^{131} has been added is dissolved in this way, substantially all the radioactivity is coprecipitated with the silver chloride. Portions of the irradiated tellurium gave large amounts of I^{131} in the silver chloride precipitate. Since the tellurium residue from the pile unit contained little I^{131} and all other fractions except the final product contained not more than 10% of the radioactivity of the final product, we believe the yield to be 85–95% of the I^{131} originally present.

The exposures of the operators (two) during the working up of a unit by the method is minimized by the use of lead bricks, long handled tongs and rapid handling of materials. On pocket ionization chambers 0.04–0.06 roentgen was registered on the day of processing a unit. This is well below the allowed maximum of 0.1 roentgen per day. Film badges[2] worn during the week including the day of the preparation showed less than ten per cent. of the permitted maximum exposure for the week.

The irradiated tellurium units used in this investigation were supplied by the Clinton Laboratories and obtained on allocation from the U. S. Atomic Energy Commission.

This work was supported by a grant from the American Cancer Society, recommended by the Committee on Growth of the National Research Council.

[2] Film Badge Service, Tracerlab, Inc., Boston, Mass.

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RECEIVED MARCH 11, 1948

Preparation of *t*-Butylamine

BY D. E. PEARSON, J. F. BAXTER AND K. N. CARTER

Tertiary butylamine has been made by the hydrogenation of 2,2-dimethylethylenimine[1] and by the reaction of *t*-butylmagnesium chloride and methoxyamine.[2] Simple hydrolysis of the readily obtainable *t*-butylurea (E. K.) with aqueous alkali is too slow for satisfactory use. Smith and Emerson[3] accomplished the hydrolysis indirectly by reaction with phthalic anhydride and subsequent hydrolysis of the resulting phthalimide. It has now been found that the amine is readily prepared from *t*-butylurea by saponification in aqueous ethylene glycol solution. The procedure shown has been checked according to "Organic Syntheses" recommendations.

$$(CH_3)_3CHNHCONH_2 + 2NaOH \longrightarrow (CH_3)_3CNH_2 + NH_3 + Na_2CO_3$$

Procedure

A 1-liter round-bottomed, S. T. flask was equipped with an upright condenser, and a glass tubing was led from the top of the condenser to a small flask immersed in ice-water. The trap was unnecessary, if sufficiently cold water was used in the condenser. The flask was charged with sodium hydroxide (60 g., 1.5 moles) dissolved in 75 cc. of water, *t*-butylurea (70 g., 0.6 mole) and 225 cc. of ethylene glycol (practical grade), and the mixture refluxed for four hours. The liquid temperature was 115° when refluxing started and fell to 86° at the end. The *t*-butylurea gradually dissolved, and a gelatinous mass of sodium carbonate was formed. Shorter reflux gave lower yields; longer periods gave no increase. Lower concentrations of ethylene glycol gave proportionately lower yields. The flask was then cooled, equipped for distillation and the fraction boiling at 40–60° was collected in an ice-cooled receiver. The crude amine, including any in the trap, weighed 37–39 g. It was dried with 5–7 g. of solid sodium hydroxide. If a lower, aqueous

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[1] Karabinos and Serijan, THIS JOURNAL, **67**, 1856 (1945); Campbell, Sommers and Campbell, *ibid.*, **68**, 140 (1946).

[2] Jones, *J. Chem. Soc.*, 781 (1946).

[3] Smith and Emerson, THIS JOURNAL, **67**, 1862 (1945).

layer formed, it was removed in a small separatory funnel, and the amine was redried with fresh sodium hydroxide for eight to ten hours. The filtered amine was then distilled through a short, Vigreux column into an ice-cooled receiver equipped with a soda-lime tube. The yield was 31–34 g. (71–78%), b. p. 44–46°, m. w. 73 by glass electrode titration. Double quantities also were used with no significant change in yields. It is suggested that the procedure, with obvious modifications in details, is probably applicable to the preparation of *t*-amylamine from *t*-amylurea.

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RECEIVED FEBRUARY 12, 1948

Colored Complexes of Tungsten Hexafluoride with Organic Compounds

BY HOMER F. PRIEST AND WALTER C. SCHUMB

It was observed in the course of a study of the reduction of tungsten hexafluoride by organic compounds that when the hexafluoride was dissolved in certain of these compounds deep colors were produced. Apparatus was devised in which tungsten hexafluoride could be added as liquid to a sample of the organic liquid, an atmosphere of dry air being maintained above the solution. Solutions were also prepared quantitatively by distilling the pure hexafluoride into a known weight of organic solvent contained in weighed cylindrical comparison tubes for a Coleman spectrophotometer (Model 14). The tubes could be sealed off and reweighed so as to obtain the weight of added fluoride. Spectrometric traces were made, all values being obtained by use of the potentiometer drum rather than by direct galvanometer deflection.

TABLE I

EFFECT OF DISSOLVING TUNGSTEN HEXAFLUORIDE IN ORGANIC LIQUIDS

Solvent	Color of solution	Effect of cooling to −78°	Effect of dilution	Remarks
Benzene	Red	White crystals	Orange–yellow	
Toluene	Red	None	Orange–yellow	Color retained when frozen
Acetone	Red	None	Orange	Color intensified on standing
Cyclohexanone	Red	None	Orange	Some intensification on standing
Ethyl alcohol	None	White crystals		
Carbon tetrachloride	None	White crystals		
sym-Tetrachloroethane	None	Solidified		
n-Decane	None	Solidified		
Cyclohexane	None	Solidified		
Diethylcarbitol	Violet–brown	None	Brown	
Diethyl ether	Violet–brown	None	Brown	
Dioxane	Pale red	White solid	Orange	Some crystals deposited

The tungsten hexafluoride had been prepared from metallic tungsten and fluorine, followed by redistillation to eliminate any WOF$_4$ which might be present. Analysis of the hexafluoride showed it to be better than 99% pure. The organic solvents employed were of carefully selected purity; thus, analytical reagent grade, thiophene-free benzene; reagent grade acetone, toluene, *sym*-tetrachloroethane, ethyl alcohol and diethyl ether; Eastman Kodak Co. pure grade cyclohexane, cyclohexanone, and *n*-decane; dioxane free from unsaturated compounds. Most of the liquids were dried over anhydrous calcium sulfate and redistilled.

A summary of the qualitative observations made on the solution of tungsten hexafluoride in the different organic solvents is shown in Table I.

From these results it appears that the presence of certain functional groups in the solvents employed results in similar colors; thus the colors in benzene and toluene are similar, as are those in diethylcarbitol and diethyl ether. Ketones, such as acetone and cyclohexanone, give similar colors which intensify on standing and may be due to polymerization of the ketone rather than to the formation of a complex with the hexafluoride. Chlorinated solvents and hydrocarbons containing no functional groups give no colors. Alcohol also gives no color, but it is to be expected that alcoholysis of the hexafluoride could interfere with complex formation.

Because of the intense color given by benzene, a quantitative study was made of this solvent. At concentrations above 0.04 molar the color appeared to remain stable for several weeks. Solutions were made up at three concentrations of tungsten hexafluoride, 0.048, 0.118 and 0.121 molar, and the optical densities of these solutions were measured at 520, 550, 560 and 570 μ. A plot of optical density *vs.* concentration of tungsten hexafluoride gave straight lines, indicating that the colored material follows Beer's law, and that the concentration of the colored complex is directly proportional to the tungsten hexafluoride concentration. By absorption measurements carried out on four solutions of tungsten hexafluoride and benzene in carbon tetrachloride, results were obtained which led to the conclusion that the concentration of the colored complex also varies directly as the benzene concentration, from which fact it is concluded that the complex contains one molecule of benzene per molecule of the hexafluoride, WF$_6$·C$_6$H$_6$.

It has been noted previously by others,[1] as well

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[1] See, for example, Fischer, *Z. anorg. Chem.*, **81**, 170 (1930); Kalischer, "Zur Kenntnis der Halogenide des höherwertigen Wolframs und Molybdäns," Berlin, 1902; Roscoe, *Ann.*, **162**, 351 (1872).