NOTES

t-Butyl Trichloroacetate

By Warner E. Scovill, Robert E. Burk and Herman P. Lankelma

Of the four butyl esters of trichloroacetic acid only the tertiary isomer has not been described. We find that it is readily prepared in 95% yield from trichloroacetyl chloride in the presence of pyridine, following the method used by Bryant and Smith[1] in the preparation of the corresponding acetate. No solvent was added, however, and heating was unnecessary. It was purified by distillation at 1 mm. pressure.

It may also be prepared from trichloroacetic acid and isobutylene, following the method used by Timofeev and Andreasov[2] for the preparation of the corresponding amyl ester. The isobutylene is rapidly absorbed in the trichloroacetic acid at 60°, the absorption being complete in three hours. The resulting brown, oily liquid crystallized on cooling with ice. It recrystallized well from both pentane and methanol at 0°, a yield of 80% being obtained from the former.

The following physical properties were determined: m. p. 25.5°; b. p. 37° at 1 mm. pressure; d^{25}_4 1.2363; n^{25} 1.4398.

Anal. Calcd. for $C_6H_9O_2Cl_3$: C, 32.83; H, 4.13; Cl, 48.46. Found: C, 32.50; H, 4.05; Cl, 48.65.

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(1) Bryant and Smith, This Journal, **58**, 1014 (1936).

(2) Timofeev and Andreasov, *Chem. Zentr.*, **96**, *II*, 1652 (1925).

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An Apparatus for the Manipulation of Sodium Triphenylmethide Reagent

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Sodium triphenylmethide in ether solution is a useful reagent to bring about certain condensation reactions.[1] The present note describes a simple apparatus[2] for the manipulation of the reagent permitting the withdrawal of analyzed aliquots from a single large scale preparation of the reagent so that a series of relatively small scale experiments may be carried out conveniently.

The analyzed solution of sodium triphenylmethide, prepared[3] in a two-liter bottle from one mole of triphenylchloromethane,[4] is divided into desired aliquots by means of the buret assembly illustrated in Diagram I, the essential features of which are a mercury-filled pressure release and a three-way stopcock, connected as shown. The system is swept with nitrogen[5] before use. In operation, a constant pressure of nitrogen is maintained in the system sufficient to push the reagent from the storage bottle to the buret. Transfer of the reagent from the storage bottle to the buret is effected by adjusting stopcock A to position (a), applying a positive pressure on the storage bottle while the buret communicates with the atmosphere. Transfer is stopped by adjusting stopcock (A) to position (b), equalizing the pressure between the storage bottle and the buret. The buret may now be drained into the receiver, previously filled with nitrogen.

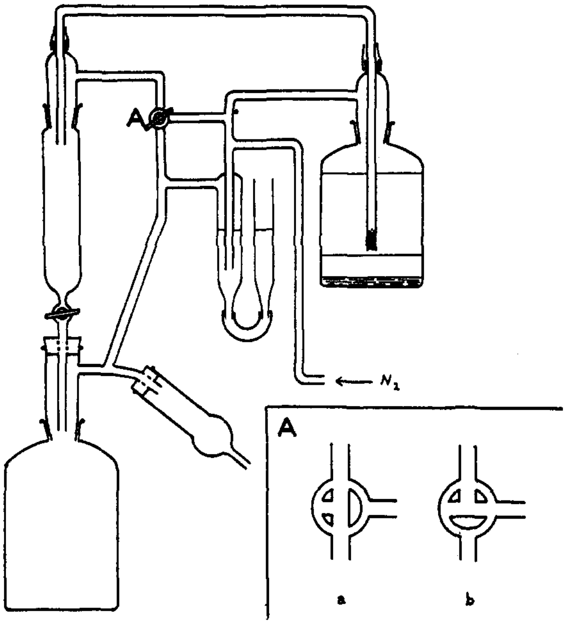

Fig. 1.

It should be noted that this apparatus maintains the reagent at all times under a small positive pressure of nitrogen preventing contact with moisture or oxygen.

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(1) See especially, Hauser and Hudson, "Organic Reactions," John Wiley and Sons, New York, N. Y., 1942, Chapter 9.

(2) An apparatus for analytical purposes has been described by Corwin and Ellington, This Journal, **64**, 2098 (1942).

(3) Hauser and Hudson, "Organic Syntheses," John Wiley and Sons, New York, N. Y., 1943, Second Coll. Vol., 609 (Note 3).

(4) Hauser and Hudson, "Organic Syntheses," John Wiley and Sons, New York, N. Y., 1943, Vol. 23, p. 102.

(5) Commercial nitrogen was used without purification with the sodium triphenylmethide reagent; see Morton and Richardson, This Journal, **62**, 123 (1940).

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Photochemical Destruction of Vitamin B_2 in Milk

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The results of experiments carried out in March, 1944, on the destruction of riboflavin in