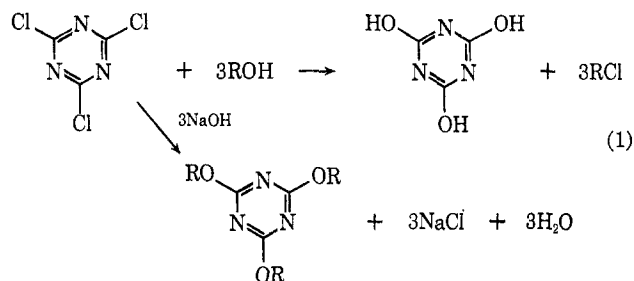


- (1) J. Liebig, *Ann. Pharm.*, **10**, 1 (1834).
- (2) P. Klaszon, *J. Prakt. Chem.*, **34**, 152 (1886).
- (3) (a) J. R. Dudley, J. T. Thurston, F. C. Schaefer, D. H. Hansen, C. J. Hull, and P. Adams, *J. Amer. Chem. Soc.*, **73**, 2986 (1951); (b) J. R. Dudley, J. T. Thurston, F. C. Schaefer, C. J. Hull, D. H. Hansen, and P. Adams, *ibid.*, **73**, 2999 (1951); (c) J. R. Dudley, U. S. Patent 2,510,564 (1950); (d) A. J. Matuszko and M. S. Chang, *J. Org. Chem.*, **27**, 677 (1962).

TABLE I
HYDROCHLORINATION OF PRIMARY, SECONDARY, AND
TERTIARY ALCOHOLS WITH CYANURIC CHLORIDE^{a,b}

Alcohol (mol)	Cyanuric chloride (mol)	Base (mol)	% yield, RCI
CH ₃ OH		NaOCH ₃	
2.5	0.5	0.5	92
3.0	0.5		72
C ₂ H ₅ OH			
5.4	0.5		70
<i>n</i> -C ₃ H ₇ OH			
4.2	0.5		65
<i>n</i> -C ₄ H ₉ OH			
3.4	0.5		41
		NaOBu	
2.5	0.5	0.5	34
<i>t</i> -C ₄ H ₉ OH			
3.4	0.5		56
Allyl alcohol			
7.8	0.5		43
<i>n</i> -C ₅ H ₁₁ OH			
4.0	0.5		44
2-C ₅ H ₁₁ OH			
1.7	0.73		29
3.4	0.50		57
3-C ₅ H ₁₁ OH			
0.85	0.49		38
C ₆ H ₅ CH ₂ OH			
3.0	0.5		71

^a The reaction temperature is the boiling point of the alcohol and the reaction time is 1–1.5 hr. ^b The chlorides were identified by their ir and nmr spectra.



lides as seen for the case of methanol and *n*-butyl alcohol in Table I. The results of Table I also indicate that cyanuric chloride can be conveniently used to hydrochlorinate primary, secondary, and tertiary alcohols. 2- and 3-pentanol are hydrohalogenated to their respective chlorides without isomerization as is not true for zinc chloride–HCl hydrochlorination (Table II). Thionyl chloride–pyridine⁴ also gives no isomerization as shown for 2-pentanol in Table II.

Experimental Section

A typical preparation involves heating the alcohol (2–20 mol) to 10–20° below its boiling point and then slowly adding powdered cyanuric chloride (1 mol). A Dry Ice trap should be connected *via* a rubber tube to the top of the reflux condenser in order to trap the low boiling chlorides. After the addition (*ca.* 1–1.5 hr), the reaction mixture is cooled, filtered, and distilled. If complete conversion to the chloride is desired,

TABLE II
HYDROCHLORINATION OF 2-PENTANOL USING VARIED METHODS

ROH	Cyanuric chloride	Mol ZnCl ₂	HCl (concd)	SOCl ₂	Pyridine	Temp, °C	Time, hr	% yield	Isomer, %	
2-Pentanol									2-Chloro	3-Chloro
3.4	0.5					117	1	57	100	
1.0		2.0	2.0			78	1–2	68	38	62
0.547				0.848	0.552	5–10	1	48	100	
0.25			0.50			86–95	1–2	27	76	24

completely ignored the hydrochlorination reaction of the alcohols.

The results of the investigation indicate that cyanuric chloride under the appropriate conditions can be used as a convenient hydrochlorinating reagent for alcohols giving no isomerization as is also true with other S_Ni reagents.^{4–9} Cyanuric chloride has the advantage that it can be conveniently handled and requires no added base such as sodium alkoxide or pyridine.^{4–6} The alcohol can be completely converted to the chloride by using an excess of cyanuric chloride under anhydrous conditions. The crude chloride is simply isolated by filtration and then purified by distillation. The presence of sodium hydroxide changes the reaction so that trialkylcyanurates are produced.³ The presence of sodium alkoxide has little effect on the reaction to give alkyl ha-

excess cyanuric chloride should be added. See Table I for molar quantities used.

Registry No.—Cyanuric chloride, 108-77-0; 2-pentanol, 6032-29-7.

Base-Induced α -Sulfonylation of Aryl Alkanesulfonates

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In the course of our investigations utilizing sulfur-stabilized carbanions for synthetic purposes,¹ it has been

(4) F. C. Whitmore and F. A. Karnatz, *J. Amer. Chem. Soc.*, **60**, 2536 (1938).

(5) E. S. Lewis and C. E. Boozer, *ibid.*, **74**, 308 (1952).

(6) L. H. Sommer, H. D. Blankman, and P. C. Miller, *ibid.*, **76**, 803 (1954).

(7) K. L. Oliver and W. G. Young, *ibid.*, **81**, 5811 (1959).

(8) K. B. Wiberg and T. N. Shryne, *ibid.*, **77**, 2774 (1955).

(9) S. J. Rhoads and R. E. Michel, *ibid.*, **85**, 585 (1963).

(1) (a) W. E. Truce and F. E. Roberts, *J. Org. Chem.*, **28**, 961 (1963); (b) W. E. Truce and L. W. Christensen, *Tetrahedron*, **25**, 181 (1969); (c) W. E. Truce and D. J. Vreneur, *J. Org. Chem.*, **35**, 1226 (1970); (d) H. Fukuda, F. F. Frank, and W. E. Truce, *ibid.*, **28**, 1420 (1963).