Methanolic Base, 0.5N

Product Specification

Methanolic Base, 0.5N (metallic sodium in methanol) is particularly useful for transesterifying triglycerides, cholesterol esters, and phospholipids. The reagent converts free fatty acids to sodium salts.

Applications/Benefits

Transesterification of triglycerides, cholesteryl esters, phospholipids. Sequence of esterification is triglycerides → diglycerides → monoglycerides → methyl esters.

Provides convenient, fast, quantitative derivatization.

Typical Procedure

This procedure is intended to be a guideline and may be adapted as necessary to meet the needs of a specific application. Always take proper safety precautions when using an esterification reagent – consult MSDS for specific handling information.

Prepare a reagent blank (all components, solvents, etc., *except sample*), following the same procedure as used for the sample.

1. Weigh 10-30mg of sample into a reaction vessel containing 1mL of organic solvent.*
2. Add 2.0mL Methanolic Base, 0.5N, and mix.
3. Heat at 70-80°C for 15-20 minutes. Allow mixture to cool to room temperature, then add 1mL water and 1mL hexane or heptane.
4. Allow phases to separate, then carefully remove upper (organic) layer and dry it over anhydrous sodium sulfate.
5. Analyze 1µL aliquot.

   Derivatization times vary widely, depending upon the specific compound(s) being derivatized. If derivatization is not complete, use additional reagent or reevaluate temperature/time of reaction.**

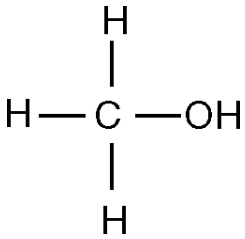
Properties

Sodium

CAS Number: 7440-23-5
Molecular Formula: Na
Formula Weight: 22.99
Appearance: light silver-white metal (tarnishes to dull gray on exposure to air)

Methanol

Structure:



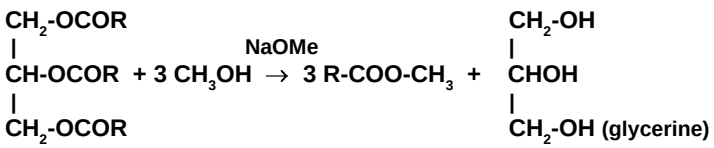
CAS Number: 67-56-1
Molecular Formula: CH_3OH
Formula Weight: 32.04
bp: 64.7°C
Flash Point: 52°F (11°C)
d: 0.791
***n*D:** 1.3290 at 20°C
Appearance: clear colorless liquid

796-0598

* Nonpolar lipids, cholesterol esters, and triacylglycerols are not soluble in reagents composed predominantly of methanol. We recommend using another solvent, such as toluene or tetrahydrofuran, to ensure reasonable reaction time. Chloroform should not be used because it usually contains ethanol as a stabilizer. In the presence of sodium methoxide (NaOMe), ethanol can ethylate the lipid, or can create dichlorocarbene, which reacts with double bonds and generates dichlorocarbene derivatives.

Cholesterol esters are transesterified very slowly and may require twice the reaction time of other compounds.

**Amide-bound fatty acids, as in sphingolipids, are not affected by alkaline transesterification reagents under mild conditions. Aldehydes are not liberated from plasmalogens with basic reagents.



Adapted from (3).

Mechanism (1,2,3)

Transesterification

In transesterification, an alcohol is displaced from an ester by another alcohol (e.g., methanol) in a process similar to hydrolysis (the second alcohol is used instead of water), forming a new ester. Transesterification is an equilibrium reaction. To shift the reaction to the right, it is necessary to use a large excess of the second alcohol, or to remove one of the products from the reaction mixture. The reaction requires 3 moles of alcohol for each mole of triglyceride. Conversion is maximized if excess alcohol is used. The conversion rate also is influenced by the reaction temperature – the reaction generally is conducted near the boiling point of the alcohol. Impurities in the oil (ester) also affect conversion rates.

Toxicity - Hazards - Storage - Stability

Methanolic Base, 0.5N is a flammable, corrosive, toxic liquid. It may irritate eyes, skin, and/or the respiratory system. Recommended storage conditions for the unopened product are stated on the label. Store opened reagent in a sealed bottle or ampul. **If you store an opened container or transfer the contents to another container for later reuse validate that your storage conditions adequately protected the reagent**.

Use only in a well ventilated area and keep away from ignition sources. Moisture can hinder the reaction – it may be necessary to dry the solvents before conducting the reaction.

The reagent has a limited shelf-life, even when refrigerated, and the use of old or excessively concentrated solutions (through alcohol evaporation) often produces artifacts and a significantly lower reaction yield.

References

1. K. Blau and J. Halket *Handbook of Derivatives for Chromatography* (2nd ed.) John Wiley & Sons, New York (1993).
2. D.R. Knapp *Handbook of Analytical Derivatization Reactions* John Wiley & Sons, New York (1979).
3. *Bailey's Industrial Oil and Fat Products*, fifth edition, Vol. 5, John Wiley & Sons, New York (1995).

Additional Reading

G. Marquez-Ruiz, M. Tasioula-Margari, C.M. Dobarganes *Quantitation and Distribution of Altered Fatty Acids in Frying Fats* JAOCS, **72:** 1171-1176 (1995).

G.F. Dunlap, J.P. White, M.L. Pollak, J.T. Brumm *Fatty Acid Composition of Oil from Adapted, Elite Corn Breeding Materials* JAOCS, **72:** 981-987 (1995).

C. Wesen, L.H. Mu, P. Sundin, P. Froyen, J. Skramstad, G. Odham *Gas-Chromatographic-Mass-Spectrometric Identification of Chlorinated Octadecanoic Acids in Eel Lipids* J. Mass Spectrom., **30:** 959-968 (1995).

P.M. Yurawecz, A.A. Molina, M. Mossoba, Y. Ku *Estimation of Conjugated Octadecatriens in Edible Fats and Oils* JAOCS, **70:** 1093-1099 (1993).

Ordering Information

Description	Cat. No.
Methanolic Base, 0.5N	
10 x 1mL	**33081**
30mL	**33352**
100mL	**33080**

Microreaction Vessels with Hole Caps and Septa

pk. of 12	
1mL	**33293**
3mL	**33297**
5mL	**33299**

Books

Handbook of Derivatives for Chromatography	
K. Blau and J. Halket	**26566-U**
Handbook of Analytical Derivatization Reactions	
D.R Knapp	**23561**

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