



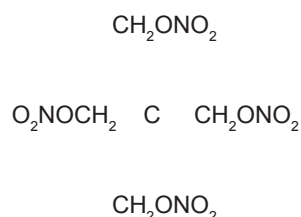
Application Note 211

Analysis of Pentaerythritol Tetranitrate on the CE440 Elemental Analyzer

Introduction

Pentaerythritol Tetranitrate (PETN) is a compound commonly used industrially in the manufacture of detonating fuse and therapeutically as a vasodilator.

6908. Pentaerythritol Tetranitrate. 2,2-Bis (nitrooxy)-methyl-1,3-propanediol dinitrate (ester) pentaerythryltetranitrate; 2,2-bis(dihydrozomethyl-1,3-propanediol tetranitrate; PETN; nitropentaerythritol; penthril; niperyt; Lentrat; Hasethrol; Peritrate; Mycardol; Nitropenton; Pentral 80; Dipentrate; Dilcoran-80; Terpat; Pentrite; Perityl; Pentritol; Pentanitrene; prevangor; Subicard; Pentryate; Vasodiatol; Neo-Corovas; Pentafin; Quintrate; Pergitral; Pentrtrate; Metranil; Peridex; Cardiacap; Angitet; Nitropenta. $C_5H_8N_4O_{12}$; mol wt 316.15. C 18.99%, H 2.55%, N 17.72% O 60.73%. Prepd by nitration of pentaerythritol: Acken, Vyverberg, U.S.D. pat. 2,370,437 (1945 to du Pont).



Tetragonal holohedra from acetone + alcohol, mp 140°, d $4^{20} 1.773$. Soluble in acetone. Practically insoluble in water (1.5 /ml); sparingly soluble in alcohol, ether. Does not reduce Fehling's solution (difference in erythryl tetranitrate). Caution: Explodes on percussion. More sensitive to shock than TNT. For medicinal purposes it is diluted with an inert ingredient, usually lactose, to prevent accidental explosions. USE: Mainly in a manufacture of detonating fuse (Primacord), a waterproof textile filled with powdered PETN. THERAP CAT: Vasodilator.

Historically the nitrogen content of this compound has been used as an indicator of purity during the manufacturing process.

The standard conditions of analysis on a CE440 Elemental Analyzer result in low nitrogen recoveries for this compound, sometimes as much as several percent nitrogen. This is believed to be the result of the formation of nitrogen oxides that are retained on the copper in the reduction tube.

The standard "fix" for nitro compounds is the addition of vanadium pentoxide as an oxygen donor plus lowering the reduction tube temperature below 600°C. For PETN, these conditions provide little improvement for the sample. The addition of an oxygen donor to an already oxygen-rich sample encourages the formation of nitrogen oxides that may be retained in the reduction tube.

Thus the successful analysis results from reducing the available oxygen to the sample; enough to form H₂O and CO₂ but not plentiful enough to easily form oxides of nitrogen. The following conditions have been established for the successful determination of nitrogen content in PETN. This procedure also ensures satisfactory determination of carbon and hydrogen content. The conditions as specified apply to the Exeter Analytical CE440 Elemental Analyzer:

- Combustion Time - 35 seconds (Resulting in the elimination of one burst of oxygen and thus a reduced oxygen condition)
- Combustion Temperature - 995°C
- Reduction Temperature - 610°C
- Tin Capsules used for sample containment
- Broad Spectrum Combustion Aid (P/N 650-00008) added to sample, 8-1 mg per sample. Combustion Aid loaded on top of sample in tin capsule; capsule sealed; then agitated to mix the sample with combustion aid.
- Sample size of 160 micrograms used. If weighed to hydrogen content, 400 micrograms would typically be specified, which is excessive for this sample type. Calibration standards should be weighed to the correspondingly lower range also.
- 1 The Merck Index, 9th edition, Merck & Co., Rahway, NJ, 1976.

The logo for Exeter Analytical, consisting of the letters 'EAI' in a large, bold, serif font. A horizontal red line passes through the middle of the letters.

EXETER ANALYTICAL

University of Warwick Science Park, The Venture Centre, Sir William Lyons Road,
Coventry CV4 7EZ. United Kingdom

Tel: +44 (0)24 76323223 Fax: +44 (0)24 76323221 Email: sales@exeteranalytical.co.uk