



Application Note 221

Kjeldahl Replacement: Nitrogen Analysis on the CE440 Elemental Analyzer

The determination of the Nitrogen content in agricultural and biological products has conventionally been done with the classical Kjeldahl technique. Many laboratories have replaced the rigorous Kjeldahl method with the Model CE440 Elemental Analyzer because it provides an easy and accurate alternative for the determination of Nitrogen.

The Kjeldahl procedure involves sulfuric acid digestion of the sample followed by direct titration. The purpose of this digestion procedure is to convert Amino-N compounds to ammonium bisulfate. Caustic alkali is then added and a distillation is performed. The result of this distillation, ammonia, is finally transferred into a boric acid solution and titrated directly, thus yielding the nitrogen content.

Depending upon the sample type, the digestion procedure may have to be modified. Problem compounds include:

- 1) Heterocyclic Nitrogen Compounds
Heterocyclic aromatic compounds with nitrogen in the ring require the presence of mercury and a digestion time of 1 hour at 370°C to ensure the cleavage of the C-N-C bond.¹
- 2) Compound with N-O Linkages
Aromatic nitro compounds can be reduced to amines after being subjected to zinc and hydrochloric acid.¹
- 3) Compound with N-N Linkages
Hydrazide compounds containing the N-N linkage are subjected to reduction by red phosphorus and hydriodic acid.¹

The above reduction procedures require a minimum reaction time of ten minutes; in some cases a reaction time of up to one hour before the normal digestion/distillation process of the Kjeldahl can be carried out. The complete analysis can take almost two hours.

The Model CE440 Elemental Analyzer will determine total nitrogen concentrations in solids or liquids, organic and inorganic compounds automatically, within 5 minutes. In operation, sub-gram sample quantities of foods, feeds, peptides, plastics, or proteins are automatically injected into a combustion chamber and converted to oxides of nitrogen. These oxides are then converted to molecular nitrogen in a reduction chamber. The sample gas is then passed through a thermal conductivity cell where the nitrogen content is determined by thermal conductivity difference against a Helium reference.

The CE440 eliminates the tedious, time-consuming reduction/digestion/distillation processes of the Kjeldahl technique, along with noxious fumes and hazardous waste disposal.

A comparison study of the combustion technique of the CE440 versus Kjeldahl was requested by the U.S. Department of Agriculture. The USDA submitted three samples for the study: Alfalfa, Corn Grain and Corn Stover. Samples from several other institutions were also included.

Using the Model CE440 equipped with a single sample injector, samples in the range of 3 mg to 10 mg were automatically analyzed without requiring any special preparation. See Table I for the results.

Table I - Analysis of Agricultural Products

	%N CE440	%N Kjeldahl		%N CE440	%N Kjeldahl
Alfalfa	1.27	1.30	Pecan Tree 40	2.52	2.46
	1.33			2.50	
Corn Grain	1.44	1.48	Soybean Standard	4.56	4.40
	1.42			4.55	
Corn Stover	0.44	0.48	Malaysian Rubber A	0.30	0.27
	0.45			0.29	
Pecan Tree 9	2.49	2.56	Malaysian Rubber B	0.36	0.33
	2.43			0.35	
Pecan Tree 22	2.91	2.90	Malaysian Rubber C	2.28	2.20
	2.88			2.29	

The data indicate that the combustion technique of the CE440 compares favorably with the Kjeldahl method; while providing a safe and timely alternative to the classical determination.

1 T.S. Ma, R.C. Rittner, Modern Organic Elemental Analysis, Marcel Dekker, Inc., NY, NY 1979.



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