

Analyses of Permanent Gases, Light Hydrocarbons, and Light Polar Compounds, Using Packed Column GC

GC columns containing Carboxen-1000 packing separate mixtures of permanent gases (nitrogen, hydrogen, oxygen/argon, carbon monoxide, carbon dioxide) and C1-C3 light hydrocarbons (methane, acetylene, ethylene, ethane, propyne, propylene, propane). A 15' x 1/8" column will separate the permanent gases and C1 and C2 hydrocarbons in less than 25 minutes. A 2' x 1/8" column provides excellent resolution of the C1-C3 hydrocarbons in the absence of permanent gases. These one-column analyses are more convenient than the two-column procedures often used to separate permanent gas/light hydrocarbon mixtures.

Key Words:

- permanent gases • light hydrocarbons
- gas-solid chromatography • formaldehyde

Analysts have used a variety of combinations of packed GC columns to separate mixtures that include permanent gases (nitrogen, hydrogen, oxygen/argon, carbon monoxide, carbon dioxide) and C1-C3 hydrocarbons (acetylene, ethane, ethylene, methane, propane, propylene, propyne). A one-column procedure, however, is easier to set up and to duplicate than two-column analyses. Carboxen™-1000, a spherical carbon molecular sieve, combines analytical capabilities previously requiring two packings (Carbosieve™ S-II and Carbosieve G). The material efficiently separates permanent gases, C1-C3 hydrocarbons, and permanent gas/light hydrocarbon mixtures. In addition, Carboxen-1000 columns can be used for analyses of more polar light compounds, such as formaldehyde and sulfur dioxide.

Carboxen-1000 particles have a mean pore diameter larger than that of the Carbosieve packings (70Å, versus 39Å) and a distribution of macropores (diameter >500Å), mesopores (20-500Å diameter), and micropores (diameter <20Å) optimized to provide effective kinetics for GSC analyses. This pore size distribution, combined with a large surface area (more than 1200m²/g, versus

1000m²/g for Carbosieve S-II), ensures the Carboxen-1000 packing will provide chromatographic analyses with retention times similar to those for the Carbosieve packings, but with greater efficiency (Table 1).

A 15' (4.5m) column containing 60/80 mesh Carboxen-1000 packing effectively separates the permanent gases and C2 hydrocarbons (Figure A). Hydrocarbons separate by degree of unsaturation, with saturated compounds eluting last. Samples containing no C2 compounds can be analyzed in only 15 minutes. Higher concentrations of oxygen relative to nitrogen, as in air samples, also can be resolved under these conditions. If the separation of oxygen and nitrogen is unnecessary, a 5' (1.5m) column containing 45/60 mesh material will separate the C2 hydrocarbons from oxygen/nitrogen, carbon monoxide, carbon dioxide, and methane. Under the conditions used for Figure A, argon (approximately 0.9% of an air sample) will not separate from oxygen. If it is necessary to separate these two gases, much lower temperatures (-70°C to -80°C) are required.

A 2' (0.6m) column containing 45/60 mesh packing will separate C1-C3 hydrocarbons that are not admixed with permanent gases (Figure B). You also can monitor trace hydrocarbons on this short column (Figure C).

Figure A. Permanent Gases and C2 Hydrocarbons Resolved

Column: 60/80 Carboxen-1000, 15' x 1/8" SS (2.1mm ID)
Cat. No.: 12390-U (general configuration)
Oven Temp.: 35°C (5 min) to 225°C at 20°C/min
Carrier: helium, 30mL/min
Det.: TCD
Inj.: 0.6mL Scott gas mix (Cat. No. 2-3437) with oxygen added, 1% each analyte, 4-6µg each analyte (except N₂) on column

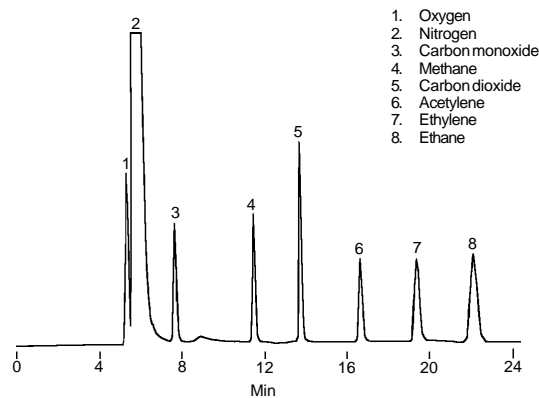


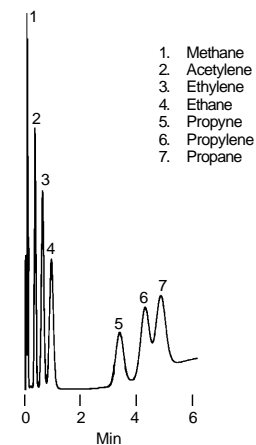
Table 1. Carboxen-1000 Columns Are More Efficient Than Carbosieve Columns

Analyte	Carboxen 1000 60/80 mesh		Carbosieve S-II 100/120 mesh		Carbosieve S-II 60/80 mesh	
	plates/foot	k'	plates/foot	k'	plates/foot	k'
CO ₂	280	12	280	12	136	11
Ethane	247	13	197	13	66	12

The surface chemistry of Carboxen-1000 particles also makes the material useful for analyses of more polar compounds, such as formaldehyde in formalin solutions and sulfur dioxide in air (Figure D). These analyses previously were performed using Carbosieves S and B, materials that have been unavailable for years.

Figure B. C1-C3 Hydrocarbons

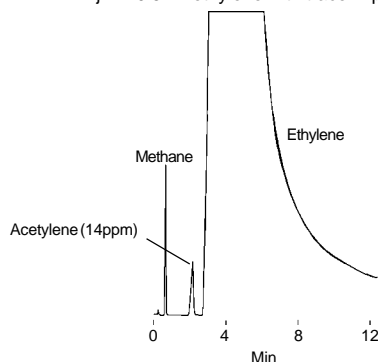
Column: **45/60 Carboxen-1000, 2' x 1/8" SS (2.1mm ID)**
 Cat. No.: **12370-U** (general configuration)
 Oven Temp.: 165°C (1 min) to 220°C at 16°C/min
 Carrier: helium, 60mL/min
 Det.: FID
 Inj.: 0.6mL Scott gas mix (Cat. No. 2-3470), 15ppm each analyte, ~10ng each analyte on column



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Figure C. Trace Acetylene in Ethylene

Column: **45/60 Carboxen-1000, 5' x 1/8" SS (2.1mm ID)**
 Cat. No.: **12380** (general configuration)
 Oven Temp.: 160°C
 Carrier: helium, 30mL/min
 Det.: FID
 Inj.: 0.5mL ethylene with trace impurities

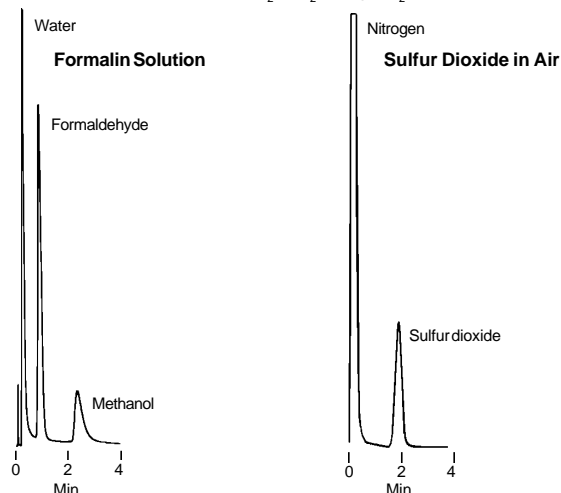


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Figure D. Polar Compounds

Column: **45/60 Carboxen-1000, 2' x 1/8" SS (2.1mm ID)**
 Cat. No.: **12370-U** (general configuration)
 Oven Temp.: 150°C (formalin) or 190°C (SO₂)
 Carrier: helium, 30mL/min
 Det.: TCD
 Inj.: 0.2µL formalin solution or 1µL 1000ppm SO₂ in N₂, ~1µg SO₂ on column



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We recommend Carboxen-1000 columns for efficient, reproducible analyses and accurate peak quantification in GSC analyses of permanent gases, light hydrocarbons, or mixtures of these two classes of compounds. If you currently are using Carbosieve S-II columns, and wish to continue to use them, packed columns and packing are still available. Carbosieve G material is available on special request.

Ordering Information:

Carboxen-1000 in 1/8" (2.1mm ID) Stainless Steel Columns

Instrument	Col. Length / Particle Size / Cat. No.		
	2'/0.6m 45/60 mesh	5'/1.5m 45/60 mesh	15'/4.5m 60/80 mesh
General configuration*	12370	12380	12390-U
Hewlett-Packard 5700-5800	12371	12381	12391
Hewlett-Packard 5880 (9" span)	12372	12382	12392-U
Perkin Elmer 900-3920	12373	12383	12393
Varian 3700	12374	12384	12394

*Coil diameter 6" (152.4mm), arm length 8" (203.2mm)

For information about custom packed columns, refer to our catalog, or call our Sales Department.

Trademarks

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■ German Pat. No. 1935500. Patent holder — Badische Anilin- & Soda-Fabrik Aktiengesellschaft.

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