

**CFA20® TESTING PROCEDURE***updated 03/01/2003***Scope**

This procedure is intended for the analysis of Fatty Acid Cetyl Esters such as Cetyl Myristoleate, Cetyl Myristate, etc. It is used specifically for determining the Cetyl Myristoleate content of finished product and in-process streams.

Work-Up Procedure

The Cetyl Myristoleate sample should be placed in a 60 degree C oven and allowed to reach thermal equilibrium. A 0.5 gram sample and 0.1 gram Methyl Tridecanoate internal standard should then be diluted in 7.5 grams carbon tetrachloride and shot on the HP 6890 GC-FID.

Gas Chromatography

Inject 0.4 uL of the cetyl ester sample into a GC under the following conditions:

Column	105 m Rtx-2330 (10% cyanopropyl, 90% ciscyanopropyl not solvent rinsable)
Carrier Gas	Hydrogen
Flow Rate	2.8 mL/min (40 cm/s constant flow)
Injector	260 degrees C
Split ratio	100:1
Shot size	0.4 uL
Temp Profile	Start at 200 degrees C for 10 minutes then ramp 5 degrees C/min to 260 degrees C. Hold for 15 minutes.
Detector	270 degrees C FID, Data Rate 50 Mhz.

Peak Identification

The free acids, alcohols and esters of interest elute as follows:

Methyl Tridecanoate	4.8
Cetyl Alcohol	6.3
Cetyl Myristate	18.3
Cetyl Myristoleate	20.0
Cetyl Oleate	22.9

Response Ratios

Cetyl Myristate : MeOTD	1.354
Cetyl Myristoleate : MeOTD	1.354
Cetyl Oleate : MeOTD	1.356

METHOD USING A POLAR MEGABORE COLUMN

Gas Chromatography

Inject 0.2 uL of the cetyl ester sample into a GC under the following conditions:

Column	10 m HP-17 (Crosslinked 50% Ph Me Silicone), 0.53 mm ID, 2 um film
Carrier Gas	Helium @ 15 psi
Injector	250 degrees C
Temp Profile	Hold 140 degrees C for 1 minute then ramp 20 degrees C/minute to 260 deg. C
Detector	250 degrees C FID, attenuation 1 x 10 ⁻¹⁰
Integrator	ATT 2 [^] = 2; CHT SP = 1.0; AR REJ = 0; THRSH = -1; PK WD = 0.06. Total Area should equal 1-3 M.

Peak Identification

The free acids, alcohols and esters of interest elute as follows:

- Myristoleic acid @ 2.5 min.
- Cetyl alcohol @ 3.0 min.
- Cetyl laurate @ 7.5 min.
- Cetyl myristate @ 8.2 min.
- Cetyl myristoleate @ 8.8 min.
- Cetyl palmitate @ 10.2 min.
- Cetyl oleate @ 13.0 min.