TABLE III

Optical Density of Mixed Carbonyl 2,4-DNP's

Conditions	Sample code	Optical density					
		430 mμ			460 mμ		
		Calculated	Found	% Error	Calculated	Found	% Error
30 min, 60C	C-H	1.121	1.105	−1.40	1.174	1.153	−1.79
	C-B	0.754	0.795	+5.45	0.894	0.923	+3.24
	B-H	0.689	0.750	+7.41	0.540	0.578	+7.03
	C-B-H	1.282	1.305	+1.80	1.304	1.328	+1.84
	Average	.....	.....	±4.02	.....	.....	±3.48
2 hr, 23C	C-H	1.121	1.115	−0.49	1.174	1.156	−1.53
	C-B	0.780	0.790	+1.28	0.909	0.923	+1.54
	B-H	0.715	0.735	+2.80	0.555	0.558	+0.54
	C-B-H	1.308	1.240	−5.20	1.319	1.323	+0.30
	Average	.....	.....	±2.44	.....	.....	±0.98
20 hr, 5C	C-H	1.121	1.124	+0.03	1.174	1.155	−1.62
	C-B	1.118	1.080	−3.22	1.142	1.140	−0.18
	B-H	1.053	1.030	−2.00	0.788	0.797	+1.14
	C-B-H	1.646	1.585	−3.10	1.552	1.525	−1.74
	Average	.....	.....	±2.09	.....	.....	±1.17

C = Crotonaldehyde
H = Hexanal
B = 2-Butanone

for acetone, crotonaldehyde and hexanal fit the latter's equations quite well and these have been used to compute the amounts of saturated and unsaturated carbonyl in heated oils.

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Gas Chromatographic Determination of Chain-Length Distribution in Fatty Acid Ethanolamides

A rapid procedure patterned after the transesterification method described by Peisker (1) for direct preparation of methyl esters from triglycerides was applied to fatty acid ethanolamides. The method is useful in studies of the relationship of chain length distribution to detergent performance.

Approximately 15 mg of fatty acid ethanolamide was weighed into a 4 in. x 7/16 in. O.D. calibrated glass stoppered test tube (Excelo), and 3 ml of methylating reagent added. The test tube was then placed in a pressure tube fabricated from ½ in. O.D. copper tubing and standard plumbing joints and the seal screwed up to finger tightness. The tube was then placed in a heating block (8 in. x 3½ in. aluminum billet drilled to accept the pressure tubes and heated electrically) for 15 minutes at 185C. Pressure tubes were removed, cooled under running water, and the glass tube removed. The contents of the tube were concentrated to 1.5 ml by immersion in a water bath and 1 ml portions of distilled water and 40–60C petroleum ether were added. The contents of the tube were shaken and the petroleum ether layer was transferred to a 7.5 x 0.8 cm round bottom sample tube with the aid of a dropper and the residue reextracted with 1 ml of petroleum ether. The two extracts were combined and methyl esters obtained by evaporation of the solvent.

Gas chromatographic separations were carried out at 170C on a "Pye" Argon chromatograph using a 4 ft 100/120 mesh Celite column containing 10% (w/w) polyethyleneglycoladipate. Chain length distribution (relative percent) was determined by cutting and weighing of individual peaks.

Results obtained for commercial samples of coco mono- and diethanolamides are shown in Table I. The results obtained by direct conversion were in good agreement with those from methylation of the isolated fatty acids. The direct conversion method is more rapid since methyl ester formation requires only 15 minutes. Careful control of the sulfuric acid content of the methylating reagent is essential for retention of liberated free amines in the aqueous phase.

TABLE I

Chain Length Distribution of Fatty Acid Ethanolamides

Carbon No.	CMEA[1]		CDEA[2]	
	a	b	a	b
8	0.97	0.67	4.62	4.30
10	4.04	4.09	6.75	6.64
12	54.97	55.28	56.83	57.13
14	18.91	19.15	16.89	16.93
16	8.87	8.60	6.99	7.18
18	12.24	12.21	7.92	7.82

[1] Coco monoethanolamides.
[2] Coco diethanolamides.
[a] By direct conversion.
[b] By methylation of isolated acids.

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