

removed and can be cleaned and reused. A schematic drawing of an injection port with the inserted injection port liner containing a peanut sample between two glass wool plugs is shown in Figure 1.

A MicroTek 2000 MF gas chromatograph equipped with a flame ionization detector and a 2 ft x 1/4 in. OD stainless steel column packed with Porapak P was used in our work. The initial temperature of the injection port was 100 C, but the temperature was programed from 60 to 180 C at 5 C/min after an initial hold period of 2 min. The helium carrier gas flow rate was 60 ml/min.

Representative chromatograms of the volatiles from ground raw peanuts and from cold pressed raw peanut oil are shown in Figure 2. Chromatograms from ground roasted peanuts and the corresponding oil are shown in Figure 3.

The presence of at least 14 and 26 compounds was indicated in raw and roasted Florigiant peanuts, respectively. Tentative identifications for 14 of the compounds are listed under Figures 2 and 3. Identifications were based on cochromatography and retention times of known compounds on the Porapak column. Most of the compounds identified in roasted (2,3) and raw peanuts (1) were identified previously. Further work using a gas chromatograph coupled with a mass spectrometer for identification of the peaks has been started.

Although the first three peaks were missing from oil sample chromatograms, the chromatograms are otherwise qualitatively similar to the chromatograms obtained from ground peanuts. The method is rapid, requiring less than one hour for preparation of the sample and completion of the analysis, and formation of artifacts appears to be nil. It provides a practical screening technique to detect the

presence of major volatiles associated with flavor, aroma, or off flavor in roasted peanuts and in oils.

DAVID F. BROWN¹

F.G. DOLLEAR

H.P. DUPUY

Southern Regional Research Laboratory²
New Orleans, Louisiana 70119

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¹National Research Council Postdoctoral Research Associate 1969-1971.

²So. Mark. and Nutr. Div., ARS, USDA.

Determination of Sodium Carboxymethylcellulose in Detergent Products

ABSTRACT

Sodium carboxymethylcellulose in detergent products reacts with phenol in a sulfuric acid media to give a colored reaction product, the absorbance of which is measured at 490 m μ . No interference was experienced from the normal constituents of detergent products. The reproducibility was found to be $\pm 0.05\%$ absolute.

Recently we needed a relatively quick and accurate method for the determination of sodium carboxymethylcellulose (CMC) in detergent products. Marier and Boulet (1) studied a colorimetric method developed by Barnett and Tawak (2) for determination of lactose in milk and

cheese. This was, in turn, based on a paper by DuBois et al. (3). It appeared likely to us that the method of Marier and Boulet could be adapted to the determination of CMC in detergents.

Subsequently we showed that such an adaptation is quite feasible. We have had no interference from the common constituents of detergent products and reproducibility of the method was found to be $\pm 0.05\%$ absolute.

The only reagents required are concentrated sulfuric acid and a phenol solution. The latter is prepared by mixing 8 g of phenol (Mallinckrodt 0008 or equivalent) and 2 g of deionized water in a small beaker, with subsequent heating on a steam bath to effect solution.

For the so-called "heavy duty" detergents presently being manufactured in this country, which contain CMC in percentages ranging from 0.5-2.5, the following procedure

TABLE I

Run No.	%CMC
1	2.15
2	2.11
3	2.11
4	2.05
5	2.15
6	2.25
7	2.17
8	2.27
9	2.15
10	2.15

TABLE II

CMC ^a originally present, %	CMC added, %	CMC found, %	Recovery, %
0.4	0.3	0.76	108.5
0.4	0.45	0.90	105.5
0.4	0.6	1.03	103.0
0.4	0.9	1.29	99.0
0.4	1.2	1.58	98.8
0.4	1.8	2.18	99.1

^aCMC = carboxymethylcellulose.

has been used.

Weigh $2.0\text{--}2.5 \pm 0.001$ g of the detergent and dissolve (warming is usually necessary) in ca. 250 ml of water. Cool and dilute to one liter. Remove a 2 ml aliquot to a test tube and add 0.1 ml of the phenol solution. Mix well, then add 6 ml of concentrated sulfuric acid solution. Again mix, then allow the solution to stand for 10 min. Measure the absorbance of the sample solution versus a blank (made by using 2 ml of water) at 490 m μ . The CMC level in the sample is obtained from a standard curve plotting absorbance versus grams of CMC. The optimum range for CMC in the test solution was found to lie between 6×10^{-5} and 16×10^{-5} g.

Table I shows data obtained by replicate determinations on the same sample. Table II shows recoveries obtained by adding known amounts of CMC to a typical household detergent product. This particular product handled the least

satisfactorily of any that we tested by this procedure and also had the lowest CMC content.

J.H. MALLORY
M.L. PORTER
Technical Center
Purex Corporation
24600 South Main Street
Wilmington, California 90744

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Selective Hydrogenation of Soybean Oil. VI. Copper-on-Silica Gel Catalysts

ABSTRACT

The preparation of copper-on-silica gel catalysts containing 15% and 20% copper is described. These catalysts can be reused three times without appreciable loss of activity. Their activity compares favorably with the highly active 5% copper-on-silica gel catalyst previously reported. Higher copper catalysts are somewhat less selective for the reduction of linolenate in soybean oil than 5% copper-on-silica gel, but these copper catalysts have greater activity, better reuse characteristics, and selectivity comparable to commercial copper-chromite catalysts.

It has been well established that copper catalysts are the most selective for the reduction of linolenate in soybean oil. The activity of copper catalysts is often influenced by the choice of support and by the method of preparation (1,2). Although copper chemisorbed on silica gel was active and selective (2), the amount chemisorbed was only 5%, so that increasing this percentage should have economic

advantages. Catalysts containing 15% and 20% copper have now been prepared. Furthermore these higher copper-on-silica gel catalysts were also prepared from copper sulfate, which is less expensive than copper nitrate previously used (2).

Enough 30% ammonium hydroxide (1.2 ml) was added to 25 ml of an aqueous solution containing 1 g of copper nitrate trihydrate to redissolve the copper hydroxide precipitate. About 2.5 g of silica gel (MS-GEL: micro-spheroidal particles with average size 54 to 65 μ , surface area = 600 sq M/g, pore volume = 1 cc/g, average pore diameter = 67 Å, supplied by Davison Chemical Division, W.R. Grace & Co.) was added to the copper-complex solution. Part of the blue copper ions was adsorbed on the surface of the gel instantly. The remainder of the copper was precipitated on the gel when the volume was made (while swirling the flask) up to 200 ml with distilled water. After the colorless supernatant was poured off, another batch of copper complex solution (1 g copper nitrate + 1.2 ml ammonium hydroxide in 25 ml) was added. While swirling the flask the volume was made up to 400 ml with

TABLE I
Hydrogenation of Soybean Oil with Copper-on-Silica Gel Catalysts^a

Catalyst	Reaction time, min	IV drop	Linolenate (alkali isomerization), %	Trans, %	Selectivity (K _{Le} /K _{Lo})
A. 15% Copper-on-silica gel (copper sulfate)	14	13	1.1	9	11
Reuse 1	14	14	1.3	9	9
Reuse 2	15	14	0.9	8	11
Reuse 3	18	14	1.4	8	9
B. 15% Copper-on-silica gel (copper nitrate)	15.5	13	1.0	9	12
Reuse 1	11.5	13	1.4	8	10
Reuse 2	13	13	1.4	10	9
Reuse 3	13	14	1.4	9	9
C. 20% Copper-on-silica gel (copper sulfate)	17	12	1.2	9	11
Reuse 1	10	13	1.5	8	9
Reuse 2	14	13	1.7	8	8
Reuse 3	16	13	1.7	8	8

^aTotal of 300 ml oil at 170 C and 30 psig; catalyst 0.3 g copper oxide.