A Comparison of Two Saponification Methods

DURING THE COURSE of investigating the unsaponifiable fraction of pork fat, it was deemed necesary to study the cold saponification technique suggested by Deuel (1951) and described in some detail by Craig et al. (1962). The efficiency of this technique was compared to a standard hot saponification method (Shone, 1962), utilizing the hydroxylamine test for esters (Hall and Shaefer, 1954).

For this study, 15 g pork or beef fat was saponified by both the hot and cold methods. When utilizing the hot method, all fat was saponified while boiling for 1.5 hr with 12% alcoholic (EtOH) potassium hydroxide. Cold saponification was carried out at room temp using sodium ethylate after the fat had been extracted from the connective tissue with redistilled diethyl ether. The fat-ether solution was mixed with the sodium ethylate and allowed to stand at room temp for 12,24,36 or 48 hr. The unsaponifiable material was extracted with redistilled diethyl ether. Material saponified by the hot method was extracted from an aqueous solution, whereas, material saponified by the cold method was extracted directly from the soap.

Saponification data for pork fat show in Table I. Each value reported is the mean from five trials, with each trial being run in triplicate. After the unsaponifiable material was extracted, the solvent was removed under vacuum at 30–35C. The drying procedure was continued until a constant wt was obtained. Good repeatability for the yield of unsaponifiable material was obtained by the hot method; whereas yields obtained by the cold method were variable. Considerable variation was also observed between triplicate samples that were saponified simultaneously by the cold method. The variation is believed to be due to difficulty in extracting the unsaponifiable material directly from the solid soap, rather than from an aqueous solution as was done in the hot procedure. An attempt was made to dissolve the soap in distilled water and then extract with ether, but this was unsuccessful.

Without using a standard curve for the hydroxylamine test, it was possible to ascertain the relative concn of ester linkages in different samples. Utilizing the test in this way, an absolute quantitative measurement is not required. The hydroxylamine efficiency values reported in Table I were based upon the relative concn of residual ester linkages and were calculated from absorbance values recorded for 15 g fat and converted to A/g unsaponifiable material. Variations in yield due to efficiency of extraction were accounted for by calculating in this manner. The A of 1 g unsaponifiable material obtained by hot saponification was assigned a value of one. All saponification efficiences are expressed relative to this arbitrary value.

TABLE I

Results of Hot and Cold Saponification

Sample No.	Time hr	Method	Yield of unsaponifiable matter (g/100 g fat)	Hydroxylamine test (OD/g)[a]	Saponification efficiency
		Saponification Data for Pork Fat			
1	1.5	Hot	0.531	0.182	1.00
2	12.0	Cold	0.308	0.201	0.91
3	24.0	Cold	0.460	0.189	0.96
4	36.0	Cold	0.465	0.148	1.23
5	48.0	Cold	0.478	0.134	1.36
		Saponification Data for Beef Fat			
1	1.5	Hot	0.455	0.099	1.00
2	12.0	Cold	0.363	0.259	0.38
3	24.0	Cold	0.384	0.182	0.54
4	36.0	Cold	0.434	0.113	0.88
5	48.0	Cold	0.397	0.108	0.91

[a] A/g unsaponifiable material. All A readings were taken at 520 mμ.

The data show that cold saponification of pork fat was essentially as efficient as the hot method, if the reaction was allowed to proceed at room temp for at least 24 hr. When the sodium ethylate was allowed to react with the extracted fat for periods of 36 hr or longer, there appeared to be a lower concn of esters.

Table I also contains the results for the saponification of beef fat by the hot and cold methods. Procedures and calculations for the beef fat study were identical to those used for pork fat. The saponification efficiency recorded for the cold procedure approached the efficiency obtained by the hot method. Upon employing the cold-procedure to obtain unsaponifiable material from beef fat, hydroxylamine A values were very similar to those recorded for pork fat. However, the hot method appeared to be more efficient for saponifying beef fat than pork fat.

These results indicate that pork fat can be saponified efficiently by the cold procedure, if a concn solution of sodium ethylate is allowed to react with the fat for 36 hr or longer. However, the extraction of unsaponifiable material from the soap appeared to be less difficult if the hot method was employed. Results also indicated that beef fat must be allowed to react with the alkali for at least 48 hr, if the saponification efficiency is to approach that obtained by the hot method.

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