(6) W. L. Chiou and G. Lam, *Int. J. Clin. Pharmacol. Ther. Toxicol.*, in press.
(7) W. L. Chiou, G. Lam, M. L. Chen, and M. G. Lee, *Res. Commun. Chem. Pathol. Pharmacol.*, **32**, 27 (1981).
(8) L. Z. Benet and R. L. Galeazzi, *J. Pharm. Sci.*, **68**, 1071 (1979).
(9) C. R. Kowarski and A. A. Kowarski, *ibid.*, **69**, 1222 (1980).
(10) M. Gibaldi, *ibid.*, **58**, 327 (1969).
(11) U. Klotz, *Clin. Pharmacokinet.*, **1**, 204 (1976).
(12) M. Rowland and G. Tucker, *J. Pharmacokinet. Biopharm.*, **8**, 497 (1980).

Win L. Chiou ˣ
Gilbert Lam
Department of Pharmacy
College of Pharmacy
University of Illinois Medical Center
Chicago, IL 60612

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Modified USP Assay of Calcium Gluceptate

Keyphrases □ Calcium gluceptate—modified USP assay □ USP assay—modification for calcium gluceptate

To the Editor:

The USP assay procedure for calcium gluceptate consists of the complexometric estimation of calcium with ethylenediaminetetraacetic acid (1). To an accurately weighed amount of calcium gluceptate (~800 mg), 150 ml of water containing 2 ml of 3 *N* HCl is added. While stirring, ~25 ml of 0.05 *M* ethylenediaminetetraacetate disodium is added from a buret. Then 15 ml of 1 *N* NaOH and 300 mg of hydroxy naphthol blue indicator are added, and the titration is continued to a blue end-point.

A similar procedure is described for the assay of precipitated calcium carbonate, calcium chloride, calcium gluconate, calcium hydroxide, calcium lactate, and calcium levulinate and for the calcium content analysis of calcium pantothenate and racemic calcium pantothenate. Although each assay calls for the addition of hydrochloric acid, this step is only necessary where the calcium salt has a limited aqueous solubility (2, 3). Since calcium gluceptate is freely soluble in water (4), we suggest that the addition of hydrochloric acid should be omitted. Table I shows that assay results are not affected by the presence or absence of hydrochloric acid.

Table I—Assay of Calcium Gluceptate in the Presence and Absence of Hydrochloric Acid

Calcium Gluceptate	Assay Value, %[a]	
	USP Method	Modified USP Method
Source A[b]	96.80 ± 0.42 (96.55–97.43)	96.82 ± 0.32 (96.54–97.10)
Source B[c]	97.02 ± 0.39 (96.51–97.42)	97.16 ± 0.21 (96.97–97.41)
Source C[d]	101.15 ± 0.08 (101.04–101.24)	100.86 ± 0.49 (100.14–101.17)

[a] Mean ± *SD*, $n = 4$; range is given in parentheses. [b] Pfanstiehl. [c] Givaudan. [d] Italsintex.

The sodium hydroxide solution and the hydroxy naphthol blue indicator can be added to the calcium gluceptate solution at the beginning of the assay; thus, it is unnecessary to interrupt titration to make these additions. The estimation of calcium with ethylenediaminetetraacetic acid using hydroxy naphthol blue as the indicator is carried out at pH 12–13 (5). The addition of 15 ml of 1 *N* NaOH solution in the official assay brings the pH to this range. In the absence of hydrochloric acid, ~10 ml of 1 *N* NaOH solution would be sufficient to bring the pH to the required range. Thus, omitting the hydrochloric acid both simplifies the procedure and enables the amount of sodium hydroxide to be reduced.

We suggest that hydrochloric acid might be omitted in the assay of other freely soluble calcium salts.

(1) "The United States Pharmacopeia," 20th rev., United States Pharmacopeial Convention, Rockville, Md., 1980, p. 107.
(2) A. Q. Butler, F. A. Maurina, and F. A. Morecombe, *J. Pharm. Sci.*, **54**, 134 (1965).
(3) R. W. Goettsch, *ibid.*, **54**, 317 (1965).
(4) "The United States Pharmacopeia," 20th rev., United States Pharmacopeial Convention, Rockville, Md., 1980, p. 1125.
(5) A. Itoh and K. Ueno, *Analyst*, **95**, 583 (1970).

R. Suryanarayanan
A. G. Mitchell ˣ
Faculty of Pharmaceutical Sciences
University of British Columbia
2146 East Mall
Vancouver, B.C. V6T 1W5
Canada

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Observed Artifacts due to Pellet Preparation in IR Spectrometry

Keyphrases □ IR spectrometry—observed artifacts due to pellet preparation □ Pellets—observed artifacts due to preparation, IR spectrometry

To the Editor:

IR spectrometry is required for identifying organic substances by most pharmacopeia and official compendia such as the USP XIX and the NF XIV (1, 2). The USP recommends: "Chemically identical substances of differing polymorphic forms often exhibit different infrared spectra when examined in the solid state. If a difference appears in the spectra, dissolve portions of both the sample and the reference standard in a suitable solvent, evaporate the solution to dryness, and repeat the test on the residues" (1). Differences resulting from polymorphism are considered to be the major reason for errors.

Certain secondary and tertiary amine derivatives such as bupivacaine, cinnarizine, and many butyrophenones have the same spectrum, in part or in whole, for their hydrochloride, hydrobromide, and free base forms when they are dissolved in methanol, ethanol, or isopropanol and if grinding with potassium bromide is prolonged.

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