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An examination of the physical properties of pure magnesium stearate

K.D. Ertel and J.T. Carstensen

School of Pharmacy, University of Wisconsin, Madison, WI 53706 (U.S.A.)

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Summary

The effect of various preparation conditions on the form of magnesium stearate prepared by aqueous precipitation was examined. None of the preparation variables had any apparent effect on the crystal habit of magnesium stearate; however, the preparation conditions did affect the amount of moisture associated with the crystals. Heating the magnesium stearate at 105 °C not only resulted in a loss of water of hydration from the crystals, but also a disruption of the crystals' 3-dimensional lattice structure. Both of these effects could be reversed by storing the dried crystals under appropriate relative humidity conditions.

Introduction

Magnesium stearate is widely used as an excipient in the production of compressed tablets, due mainly to its ability to decrease friction between the tablet's surface and die wall during the ejection process. The compound is usually effective in low concentrations; however, some care must be excercised when preparing formulations with magnesium stearate due to the compound's hydrophobic nature, which has been shown to adversely affect tablet properties such as hardness (Strickland, 1956; Shah, 1977; Lerk, 1977a; De Boer, 1978; Ragnarsson, 1979), disintegration time (Strickland, 1956; Shah, 1977; Ragnarsson, 1979), and dissolution rate (Levy, 1963; Lerk, 1977b; Shah, 1977; Iranloye, 1978; Billany, 1982). Consequently, it is desirable to optimize the amount of magnesium stearate used in a formulation, i.e. to use an amount which adequately lubricates the formulation but minimizes the adverse effects on the finished tablets. Unfortunately, the optimization process is complicated by the fact that commercial magnesium stearates exhibit significant batch-to-batch variation in their ability to function as lubricants (Hanssen, 1970; Müller, 1977a; Steffens, 1982; Rubinstein, 1983; Colombo, 1984; Hölzer, 1984).

Although attempts have been made to identify the factor(s) responsible for the batch-to-batch variation in the lubricant properties of magnesium stearate by examining commercial samples, these studies are complicated by the fact that commercial magnesium stearate is not an exact chemical entity. For this reason, a number of workers have chosen to use pure magnesium stearate, which

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Correspondence: K.D. Ertel, *Present address*: Merrell Dow Research Institute, Cincinnati, OH 45215, U.S.A.

may be prepared by precipitation from an aqueous medium according to the reaction:

$$2\ CH_3(CH_2)_{16}COOX + MgCl_2 \rightarrow [CH_3(CH_2)_{16}COO]_2Mg + 2\ XCl$$

where X is a cation such as NH_4^+ or Na^+. Both plate-shaped dihydrate and needle-shaped trihydrate forms of magnesium stearate have been prepared by this method, the hydration state being a function of the pH of the precipitation medium and the cooling rate (Müller, 1977a–c, 1982; Miller, 1982, 1985). The hydrates have different crystal habits and, more importantly, belong to different crystal systems (Müller, 1977c). The latter is a result of changes in the angle of inclination of the hydrocarbon chains relative to the plane of the magnesium atom head groups brought about by the incorporation of water into or the loss of water from the crystal lattice. An anhydrous form of magnesium stearate, prepared by drying hydrous magnesium stearate under appropriate conditions, has also been described. The hydrocarbon chains of the anhydrate are packed in a manner different from that of either of the previously mentioned hydrates (Vold, 1949; Müller, 1977a, c).

Materials and Methods

Materials

The purity of the stearic acid (99 + %, Aldrich Chemical Company, Milwaukee, WI) was verified chromatographically. Magnesium chloride (ACS grade, Drake Brothers, Menomonee Falls, WI), acetone (AR grade, Mallinckrodt Inc., St. Louis, MO), ethanol (absolute, Midwest Solvents Company of Illinois, Perkin, IL), and ammonium hydroxide (ACS grade, J.T. Baker Chemical Company, Phillipsburg, NJ) were used as received from the suppliers. Potassium bromide (spectroscopic grade, Aldrich) was dried for 2 h at 110 °C before use. A saturated solution of sodium hydroxide was prepared from sodium hydroxide pellets (reagent grade, Amend Drug and Chemical Co., Irvington NJ). Water used throughout was double-distilled from an alkaline permanganate solution in an all-glass still.

Preparation of magnesium stearate

All magnesium stearates used in this work were prepared by precipitation from an aqueous medium. After dispersing approximately 0.01 mol of stearic acid in 600 ml of water (previously heated to 90 °C), the sodium or ammonium soap of the fatty acid was formed by dropwise addition of saturated sodium hydroxide solution or ammonium hydroxide, respectively. The addition of base was continued until the pH of the soap dispersion was in the range 7–11. In most cases, precipitation was carried out at pH 9. An amount of magnesium chloride slightly in excess of that required for stoichiometric reaction with the fatty acid soap was dissolved in 100 ml of water and the resulting solution was added dropwise, with stirring, to the soap dispersion over a 20-min period. The mixture was cooled to room temperature over a period of 3–12 h, after which time the precipitate was isolated by filtration, transferred to a Soxhlet extraction apparatus, and washed for 24-h periods with acetone and water to remove any unreacted fatty acid and magnesium chloride. The washed powder was air-dried and stored under ambient conditions.

Anhydrous magnesium stearate samples were prepared immediately before use by drying hydrous magnesium stearate to constant weight at 105 °C.

Hydration behavior

To examine the hydration behavior of magnesium stearate, desiccators containing saturated solutions of the following salts were prepared (Nyqvist 1983; Weast, 1977): LiCl (11.3%), $MgCl_2$ (32.8%), K_2CO_3 (43.0%), $Mg(NO_3)_2$ (52.8%), KI (68.9%), and KBr (80.9%). The desiccators were stored at 25 ± 1 °C to maintain the relative humidities shown in parentheses. In addition, a desiccator containing phosphorous pentoxide was prepared to produce a relative humidity of 0%.

Thermal properties

The thermal properties of pure magnesium stearate samples were determined using a thermal analysis system (Series 7, Perkin-Elmer Corp., Ridgefield, CT) which included a differential scanning calorimeter (DSC) and a thermogravimetric analyzer (TGA). DSC examinations were in most cases carried out over a temperature range of 40 °C–150 °C at a heating rate of 2 °C/min, although in some cases samples were heated to temperatures as high as 200 °C at a heating rate of 10 °C/min. TGA examinations were carried out over a temperature range from 50 °C to 130 °C at a heating rate of 5 °C/min.

The appearance of the magnesium stearate during heating was observed under magnification using a polarizing microscope (Optikot-Pol, Nikon, Japan) equipped with a hot-stage (Model FP 82, Mettler Instrument Corp., Hightstown, NJ). A heating rate of 2 °C/min was maintained by a central processor (Model FP 80, Mettler).

Infrared spectroscopy

The infrared absorption spectra of stearic acid and of hydrous and anhydrous magnesium stearate samples were obtained using an infrared spectrophotometer (Model 599B, Perkin-Elmer Corp., Norwalk, CT). Ca. 1 mg of the powder to be examined was ground with 100 mg of dry potassium bromide, compressed into a pellet, and scanned from 200–4000 cm^{-1}.

X-ray analysis

Diffraction patterns for magnesium stearate were obtained using a powder diffractometer (Phillips Electronics Inc., Mount Vernon, NY). Samples were irradiated with X-rays from a copper target (Cu $k\alpha$ radiation, $\lambda = 0.15418$ nm). Two types of samples mounts were used: random powder mounts, prepared using the sample holder provided with the instrument, and ordered powder mounts, prepared by dropping an ethanolic suspension of magnesium stearate crystals onto a clean glass microscope slide and allowing the solvent to evaporate. Diffraction patterns from both types of mounts were measured from $2\Theta = 5°$ to $25°$, where Θ is the Bragg angle.

Results and Discussion

As noted in the introduction, medium pH and cooling rate are thought to be the primary factors which determine the form of magnesium stearate prepared by aqueous precipitation. Neither of these factors had any effect on the crystal habit of the magnesium stearates produced in the present work, as all of the samples consisted of plate-shaped crystals, i.e. the needle-shaped crystals described in the literature could not be isolated. The medium pH did have an effect on the amount of water associated with the crystals, however. Batches precipitated at pH 9 were shown by thermal analysis to contain an amount of bound water corresponding to a dihydrate (form A), while batches precipitated at the pH extremes (i.e. 7 and 11) contained considerably less bound water (Table I). The decreased amount of moisture associated with the latter batches may be due to irregular incorporation of water into the magnesium stearate crystal lattice during precipitation (Miller, 1982) or to the formation of a mixture of magnesium stearate dihydrate and a lower hydration state (Miller, 1985).

Earlier workers have reported that it is possible to prepare magnesium stearate trihydrate by aqueous precipitation. Despite the wide variety of precipitation conditions employed in the present study, the trihydrate could not be isolated, although several batches of magnesium stearate precipitated at pH 9 did contain slightly more water than the amount expected for a dihydrate (e.g. 2.4-hydrate). Storage of these powders over phosphorous pentoxide, however, decreased their moisture content to that of a dihydrate (Fig. 1), indicating that the excess water was present as adsorbed surface moisture.

An anhydrous form of magnesium stearate (form B) was prepared by drying the dihydrate to

TABLE 1

Effect of precipitation pH on the moisture contents of various laboratory-prepared magnesium stearates

Precipitation pH	Loss on drying (weight %)
9	5.56 ± 0.17 ($n = 4$)
9	5.71 ± 0.08 ($n = 8$)
9	5.22 ± 0.11 ($n = 3$)
9	5.54 ± 0.21 ($n = 4$)
7	2.06 ± 0.01 ($n = 3$)
11	3.42 ± 0.31 ($n = 8$)

Values are mean ± S.E.M., n denotes the number of determinations. The theoretical weight loss for a dihydrate is 5.74%

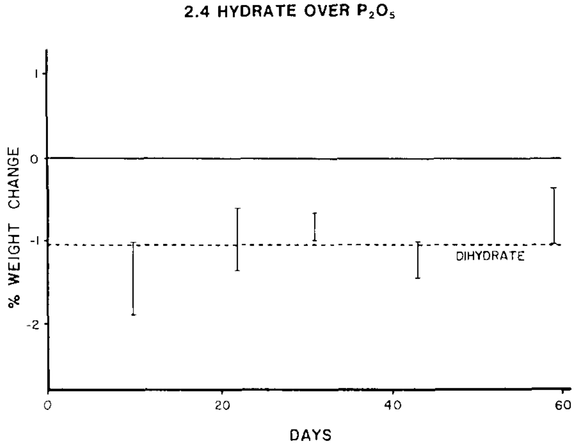

Fig. 1. Weight loss of magnesium stearate form A (2.4-hydrate) when stored over phosphorous pentoxide. Error bars represent 95% confidence limits.

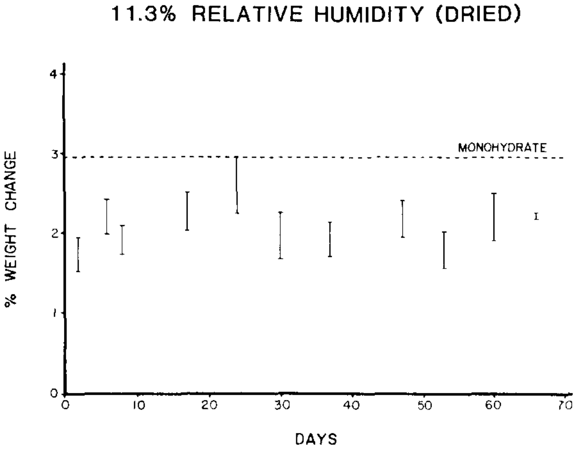

Fig. 2. Moisture uptake of magnesium stearate anhydrate (form B) when stored under 11.3% relative humidity conditions. Error bars represent 95% confidence limits.

constant weight at 105 ° C. This treatment resulted in a loss of moisture only; the plate-shaped appearance of the original dihydrate was retained. The loss of moisture was, however, accompanied by the development of striations in the crystals and a darkening of the crystals' appearance when viewed under crossed polars, indicating a loss of their anisotropic property, as has been described previously (Miller, 1985). These changes were most likely due to desolvation rather than to a polymorphic change in the crystals.

Magnesium stearate dihydrate exhibited no tendency to sorb moisture when stored over the various saturated salt solutions described earlier. This is not surprising, since magnesium stearate is classified as being non-hygroscopic (Callahan, 1982), although the compound apparently will sorb moisture when stored under relative humidities greater than approximately 85% (Callahan, 1982; Hölzer, 1985; Handbook of Pharmaceutical Excipients). Furthermore, except for the specific cases mentioned above, the dihydrate exhibited no tendency to lose moisture when stored over phosphorous pentoxide.

Unlike the dihydrate, the anhydrous form of magnesium stearate did exhibit a tendency to sorb moisture when stored over the various saturated salt solutions (Figs. 2 and 3). This moisture was present as adsorbed surface moisture when the relative humidity was less than 50%, as evidenced by the rapid and continuous weight loss observed during thermogravimetric analysis (Fig. 4). This type of moisture sorption has previously been described for anhydrous magnesium stearate (Müller, 1982). When the relative humidity exceeded 50%, however, the anhydrate rehydrated to form a trihydrate (form C). This phenomenon has not been previously described for anhydrous magnesium stearate, although other anhydrous metal soaps are known to rehydrate under appropriate conditions. The rehydration process was accompa-

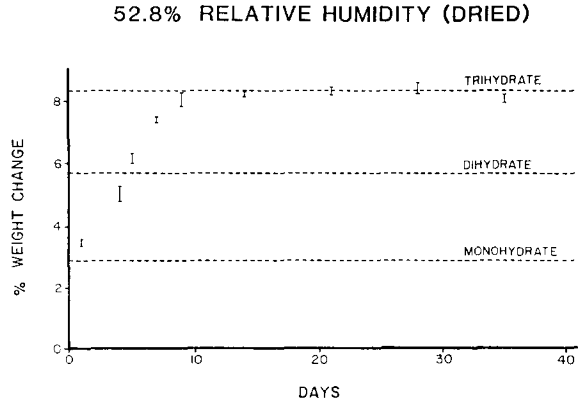

Fig. 3. Moisture uptake of magnesium stearate anhydrate (form B) when stored under 52.8% relative humidity conditions. Error bars represent 95% confidence limits.

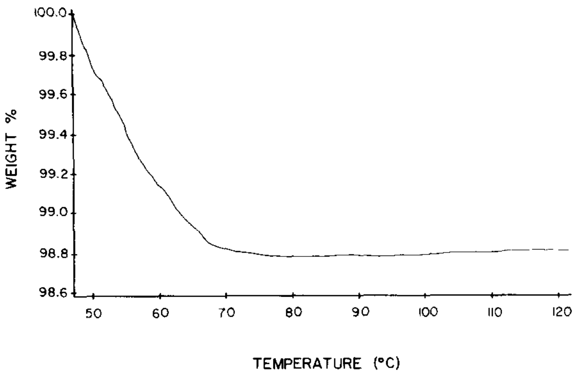

Fig. 4. Typical % weight vs temperature curve (TGA) for magnesium stearate anhydrate (form B) stored under relative humidities less than 50%. The example shown is from a sample stored under 11.3% relative humidity.

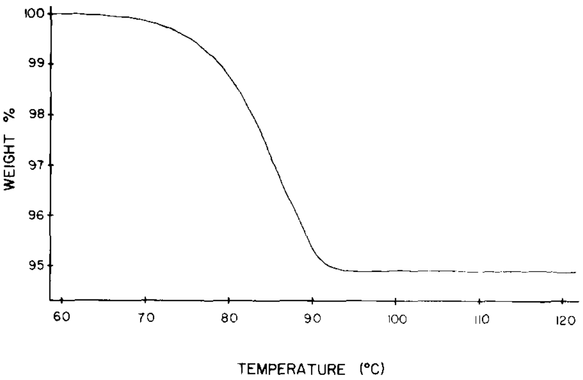

Fig. 5. Typical % weight vs temperature curve (TGA) for magnesium stearate dihydrate (form A).

nied by a significant increase in the crystal long spacing of the magnesium stearate (Table 2), indicating that a change in the packing of the hydrocarbon chains had occurred. Once formed, the trihydrate was stable at 25 ° C when stored under relative humidity conditions less than those required for its formation, i.e. less than 50%. The anhydrate could be regenerated, however, by drying the trihydrate at 105 ° C.

Thermogravimetric analysis of the dihydrate and trihydrate forms of magnesium stearate showed that the waters of hydration within a given hydration state were bound equivalently, as evidenced by the lack of shoulders in their weight loss curves (Figs. 5 and 6). The thermal behavior of all 3 forms of magnesium stearate was further characterized by DSC. Thermograms of both the dihydrate (Fig. 7) and trihydrate (Fig. 8) forms of magnesium stearate exhibited two thermal events, although the peak temperatures associated with these events were shifted to lower values for the latter form (Table 3). No thermal events were observed at temperatures greater than 130 ° C for any of the samples. Previous workers have assigned the low temperature endotherm to the loss of bound water from the crystals and the high temperature endotherm to a melting phenomenon (Müller, 1977a, c; Miller, 1985). These assignments were confirmed in the present work by TGA and hot-stage microscopy. As shown in Table 3, the enthalpic change associated with the loss of bound water from the dihydrate was significantly greater than that associated with the loss of bound

TABLE 2

Crystal long spacing (d_{001}) of magnesium stearate anhydrate (form B) as a function of relative humidity

Relative humidity (%)	d_{001} (nm)
0.0	5.023 ± 0.005 (n = 10)
11.3	4.999 ± 0.006 (n = 10)
32.8	5.034 ± 0.006 (n = 16)
43.0	5.056 ± 0.005 (n = 6)
52.8	5.270 ± 0.003 (n = 16)
68.9	5.271 ± 0.003 (n = 13)
80.9	5.304 ± 0.003 (n = 18)

Values are mean ± S.E.M., n denotes the number of determinations.

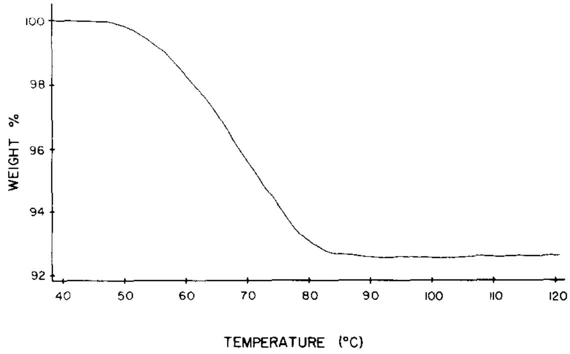

Fig. 6. Typical % weight vs temperature curve (TGA) for magnesium stearate trihydrate (form C).

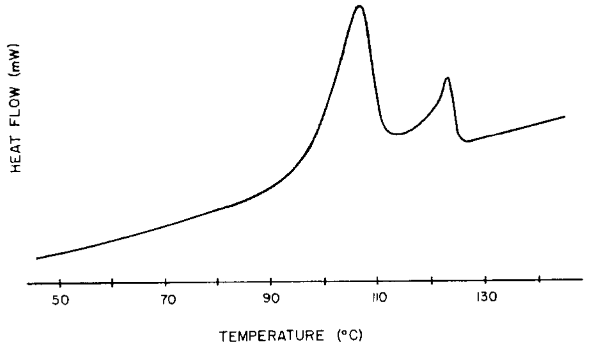

Fig. 7. Typical thermogram (DSC) produced by heating magnesium stearate dihydrate (form A) at a rate of 2° C/min.

water from the trihydrate, indicating that the bonds holding the water in the two hydration states were of different strengths, i.e. water was bound more tightly in the dihydrate than in the trihydrate. This finding is consistent with what has been reported previously for magnesium stearate dihydrate and trihydrate (Müller, 1977b). The thermogram obtained from the anhydrous form of magnesium stearate (Fig. 9) exhibited a single endothermic transition, that associated with the melting of the crystals. For the particular case shown, the lack of an endotherm corresponding to the loss of bound moisture from the crystals provides further evidence that moisture sorbed when the anhydrate was stored under relative humidities less than 50% existed as adsorbed surface moisture only.

The structural differences between the 3 hydrates alluded to earlier were confirmed by X-ray powder diffraction using randomly oriented samples (Fig. 10). Of particular interest is the region near $2\Theta = 21°$. The diffractogram of the dihydrate exhibited several distinct peaks in this region, while in the case of the anhydrate, these peaks were replaced by a single broad peak. This type of peak is known as a halo, and is indicative of a structure in which the magnesium atoms of magnesium stearate are arranged in irregularly spaced parallel planes, i.e. the three-dimensional structure of the crystal lattice has been disrupted (Vold, 1949). The diffractogram of the trihydrate again displays several distinct peaks in the region near $2\Theta = 21°$, indicating that the three-dimensional lattice structure has been restored by rehydrating the crystals.

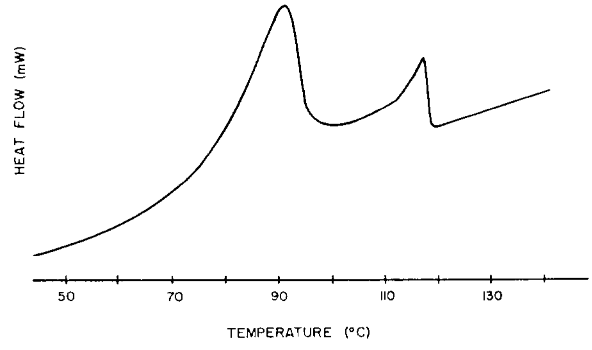

Fig. 8. Typical thermogram (DSC) produced by heating magnesium stearate trihydrate (form C) at a rate of 2° C/min.

TABLE 3

Thermal properties of the various forms of magnesium stearate as determined by DSC

Form	T_1 (°C)	ΔH_1 (J/g)	T_2 (°C)	ΔH_2 (J/g)
A[a]	100.1	229	122.5	58.6
B[b]	–	–	125.1	59.7
C[a]	82.9	146	116.7	35.1

[a] Heating rate 2° C/min.
[b] Heating rate 10° C/min.

Crystal long spacings for the various forms of magnesium stearate were calculated from data obtained from diffractograms generated using

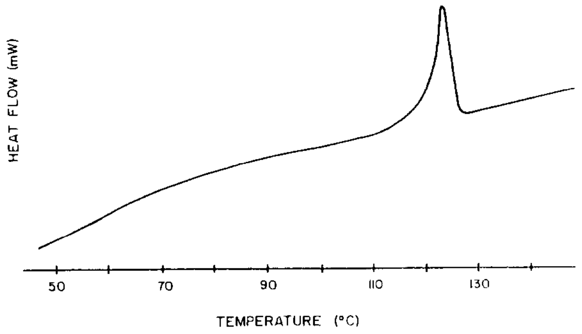

Fig. 9. Typical thermogram (DSC) produced by heating magnesium stearate anhydrate (form B) at a rate of 2° C/min. The example shown is from a sample stored under 11.3% relative humidity.

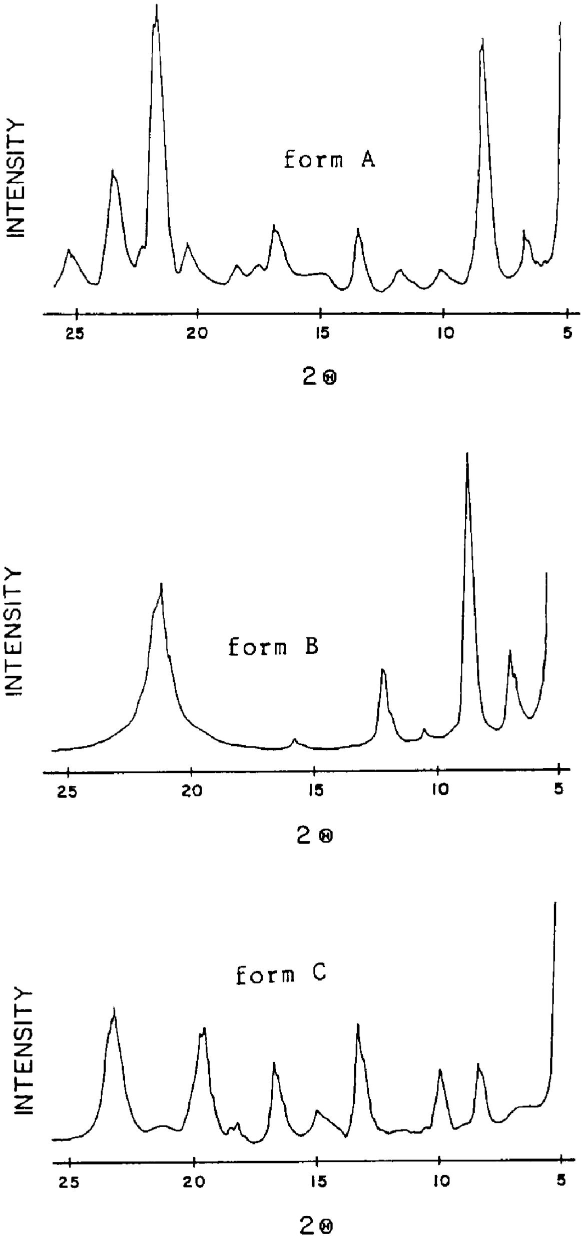

Fig. 10. X-ray diffraction patterns obtained from random powder mounts of the various forms of magnesium stearate.

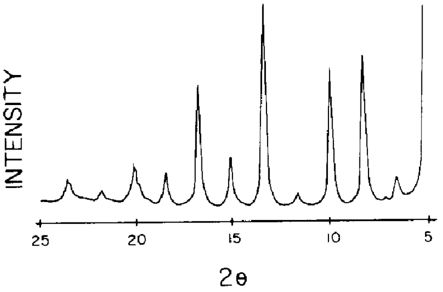

Fig. 11. Typical X-ray diffraction pattern obtained from an ordered mount. The example shown is from a sample of magnesium stearate trihydrate (form C).

ordered powder mounts, since this type of mount minimized the contributions of the other lattice spacings and made the diffraction patterns easier to interpret. A typical diffraction pattern obtained from an ordered powder mount is shown in Fig. 11. Diffraction angles corresponding to peak maxima were converted to crystal long spacings using the Bragg equation:

$$n\lambda = 2d \sin \Theta \tag{1}$$

where d is the crystal long spacing, λ is the wavelength of the X-rays, Θ is the Bragg angle, and n is the diffraction order (e.g. $d/2$, $d/3$, $d/4, \ldots,$). The long spacing calculated for the dihydrate is identical to that reported previously (Müller, 1977b, c); however, the long spacings calculated for the anhydrate and trihydrate forms are significantly greater than the values reported previously for these hydration states (Table 4). These data suggest that the crystal structures of the anhydrate and trihydrate forms of magnesium stearate isolated in the present work are different

TABLE 4

Crystal long spacings (d_{001}) of the various forms of magnesium stearate

Sample	d_{001} (nm)
Form A (via ammonium soap)	5.220 ± 0.003 (n = 20)
Form A (via sodium soap)	5.219 ± 0.003 (n = 20)
Form A (2.4 hydrate)	5.239 ± 0.004 (n = 20)
Form B	5.032 ± 0.005 (n = 10)
Form C (52.8% R.H.)	5.270 ± 0.003 (n = 16)
Form C (80.9% R.H.)	5.304 ± 0.003 (n = 18)
Anhydrate *	3.55
Dihydrate *	5.23
Trihydrate *	5.13

Values are mean ± S.E.M., n denotes the number of determinations.

* Values reported by Müller (1977a).

TABLE 5

Summary of infrared absorption data for stearic acid and the various forms of magnesium stearate.

Wavenumber (cm^{-1})	Stearic Acid	Form A	Form B	Form C
3 600–3 100 (water of crystallization)	–	s	(w)	s
3 300–2 500 (-OH stretch)	m (broad)	–	–	–
2 900 (C–H symmetric and asymmetric stretch)	m	s (doublet)	m (broad)	s (doublet)
1 700 (C=O stretch)	s	–	–	–
1 616–1 540 (COO^- asymmetric stretch)	–	s	s	s
720 (CH_2 rocking)	w (doublet)	w (doublet)	w (singlet)	w (singlet)

w, weak; m, moderate; s, strong.

from the crystal structures of corresponding hydration states isolated in earlier studies.

The difference between the anhydrate and trihydrate forms isolated in this work and in earlier studies was further demonstrated by examining the infrared (IR) absorption spectra of the compounds. The pertinent absorption data for stearic acid and the 3 hydrates of magnesium stearate isolated in this work are summarized in Table 5. Because formation of the magnesium salt of stearic acid occurred at the carbonyl end of the molecule, the observed differences between the absorption spectra of stearic acid and magnesium stearate are mainly due to changes in the absorption bands associated with the carbonyl group. For example, spectra of all forms of magnesium stearate exhibited a shift from 1700 cm^{-1} to 1615–1540 cm^{-1} in the absorption band due to the carbonyl stretch, indicating that the carbonyl group had gone from an unionized to an ionized state in forming the magnesium salt of stearic acid (Lecomte, 1950; Mehrotra, 1983). There were, however, several changes in the spectra associated with the aliphatic portion of the molecule. Of these, the absorption band appearing at approximately 720 cm^{-1} was of special interest. This particular band is due to the CH_2 rocking mode, and its appearance (i.e. as a singlet or a doublet) is related to the packing of the hydrocarbon chains within the crystal (Chapman, 1957). Bands appearing as singlets correspond to either triclinic or hexagonal packing, while bands appearing as doublets correspond to orthorhombic packing, although it has been argued that a doublet may correspond to monoclinic packing as well (Müller, 1977b). Based on the appearance of this absorption band, the hydrocarbon chains in the stearic acid crystals possessed either a monoclinic or an orthorhombic packing, which is consistent with the packing determined for stearic acid by X-ray diffraction (von Sydow, 1956). The hydrocarbon chains in the dihydrate form of magnesium stearate also existed in either a monoclinic or an orthorhombic packing, which is again consistent with the packing previously described for this species (Müller, 1977c). The triclinic or hexagonal packing determined for the trihydrate, however, is not consistent with the orthorhombic packing previously ascribed to this hydrate. The anhydrate has previously been reported to possess hexagonal packing (Vold, 1949; Müller, 1977c, 1982), which at first appeared to be consistent with the hexagonal or triclinic packing determined for the anhydrate in this work. The angle of inclination of the hydrocarbon chains calculated from the X-ray data, however, was not consistent with hexagonal packing, indicating that the hydrocarbon chains existed in a triclinic packing.

Conclusion

Based on what has been discussed above, the following conclusions were drawn:

1. The crystal habit of magnesium stearate was not sensitive to either the pH of the precipitation medium or the cooling rate, as all samples prepared consisted of plate-shaped crystals. The

needle-shaped crystals that have been reported previously could not be isolated under any conditions.

2. The pH of the precipitation medium did have an effect on the amount of moisture that was bound within the magnesium stearate crystals. Samples precipitated from media at pH 9 contained bound moisture equivalent to that expected for a dihydrate, while samples precipitated from media at higher or lower pH values contained significantly less bound moisture.

3. Drying the dihydrate at 105°C resulted in the production of an anhydrous form of magnesium stearate. The anhydrate retained the appearance of the original dihydrate; however, heating resulted in a disruption of the crystals' 3-dimensional lattice structure.

4. The dihydrate form of magnesium stearate exhibited no tendency to gain or lose moisture under any of the relative humidity conditions used in this work. The anhydrate form sorbed moisture readily, and rehydrated to a trihydrate when the relative humidity exceeded 50%. The trihydrate, like the dihydrate, did not exhibit any tendency to lose moisture when stored under any of the relative humidity conditions used in this work.

5. The dihydrate form of magnesium stearate isolated in the present work appears to be identical to previously reported dihydrates. This is not true for the anhydrate and trihydrate, as both of these forms exhibit physical properties different from those described previously for corresponding hydration states.

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