IN THE UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF DELAWARE

MERCK SHARP & DOHME CORP.,	)	
	)	
Plaintiff,	)	
	)	
v.	)	C.A. No. 15-250-SLR-SRF
	)	
AMNEAL PHARMACEUTICALS LLC,	)	
	)	
Defendant.	)	

FIRST AMENDED COMPLAINT FOR PATENT INFRINGEMENT

For its First Amended Complaint, Plaintiff Merck Sharp & Dohme Corp. alleges as follows:

NATURE OF THE ACTION

1. This is an action for patent infringement arising under the patent laws of the United States of America, Title 35, United States Code, against Defendant Amneal Pharmaceuticals LLC ("Amneal"). This action relates to Abbreviated New Drug Application ("ANDA") No. 207989 filed by Amneal with the U.S. Food and Drug Administration ("FDA") for approval to market mometasone furoate nasal spray, a generic version of Plaintiff Merck Sharp & Dohme Corp.'s (Merck's) Nasonex® drug product, prior to expiration of U.S. Patent No. 6,127,353 (the '353 patent).

PARTIES

2. Merck is a New Jersey corporation with its principal place of business at 2000 Galloping Hill Road, Kenilworth, New Jersey 07033.

3. On information and belief, Amneal is a limited liability company organized and existing under the laws of Delaware with a principal place of business at 400 Crossing Blvd., Bridgewater, New Jersey 08807.

JURISDICTION AND VENUE

4. This action arises under the patent laws of the United States of America, Title 35, United States Code, and jurisdiction is founded on Title 28, United States Code §§ 1331 and 1338(a).

5. Venue is proper in this Court under 28 U.S.C. §§ 1391 and 1400(b).

6. This Court has jurisdiction over Amneal because, upon information and belief, Amneal is a limited liability company organized and existing under the laws of Delaware.

7. Upon information and belief, Amneal is registered to conduct business with the State of Delaware and maintains as a registered agent The Corporation Trust Company, Corporation Trust Center, 1209 Orange St., Wilmington, Delaware 19801.

8. Upon information and belief, Amneal is registered pursuant to Del. Code Ann. Tit. 24, § 2540 to distribute its generic pharmaceutical products in Delaware.

9. This Court also has jurisdiction over Amneal because, inter alia, this action arises from actions of Amneal directed toward Delaware, and Amneal has purposefully availed itself of the rights and benefits of Delaware law by engaging in systematic and continuous contacts with Delaware. Upon information and belief, Amneal regularly and continuously transacts business within the State of Delaware, including by selling pharmaceutical products in Delaware, either on its own or through affiliates. Upon information and belief, Amneal derives substantial revenue from the sale of those products in Delaware and has availed itself of the privilege of conducting business within the State of Delaware.

10. Amneal has previously been sued in this judicial district without objecting on the basis of lack of personal jurisdiction and has availed itself of Delaware courts through the assertion of counterclaims and by filing suits in Delaware.

11. For these reasons, and for other reasons that will be presented to the Court if jurisdiction is challenged, the Court has personal jurisdiction over Amneal.

BACKGROUND

12. On October 3, 2000, the '353 patent, entitled MOMETASONE FUROATE MONOHYDRATE, PROCESS FOR MAKING SAME AND PHARMACEUTICAL COMPOSITIONS, duly and legally issued to Pui-Ho Yuen, Charles Eckhart, Teresa Etlinger, and Nancy Levine. The '353 patent is currently scheduled to expire on October 3, 2017, with pediatric exclusivity through April 3, 2018. The '353 patent discloses and claims novel form(s) of mometasone furoate monohydrate (also designated 9α,21-dichloro-16α-methyl-1,4-pregnadiene-11β,17α-diol-3,20-dione-17-(2'-furoate) monohydrate) and novel pharmaceutical compositions thereof. A copy of the '353 patent is attached to this First Amended Complaint as Exhibit 1.

13. Merck is the owner through assignment of the '353 patent, and is the owner of approved New Drug Application No. 20762, covering mometasone furoate monohydrate metered nasal spray that is sold under the Nasonex® trademark.

14. Merck's Nasonex® nasal spray is extremely successful and is widely used in Delaware, the United States, and throughout the world to treat diseases of the upper airways, including allergic and nonallergic rhinitis.

15. The publication *Approved Drug Products with Therapeutic Equivalence Evaluations* (commonly known as the "Orange Book") identifies drug products approved on the basis of safety and effectiveness by the FDA under the Federal Food, Drug, and Cosmetic Act (FFDCA). Merck has listed the '353 patent in the Orange Book as covering its Nasonex® nasal spray.

16. On information and belief, Amneal has filed an ANDA with the FDA for generic mometasone furoate nasal spray, 50 mcg (ANDA No. 207989). Amneal's ANDA No. 207989 allegedly contains a certification under Title 21, United States Code § 355(j)(2)(A)(vii)(IV) and Title 21, Code of Federal Regulations, § 314.95, that the '353 patent is "unenforceable, invalid, and/or not infringed." Notice of that certification, but not the certification, was transmitted to Merck on or after February 3, 2015, and received by Merck on or after February 4, 2015.

17. Amneal refused to make ANDA No. 207989 or samples of its proposed generic copy of Nasonex® nasal spray available to Merck under reasonable conditions that would allow evaluation of the ANDA and/or samples before the filing of the original complaint in this action.

18. Upon information and belief, Amneal's proposed generic copy would contain mometasone furoate in such a form that would infringe the '353 patent.

19. On information and belief, Amneal filed ANDA No. 207989 because Amneal seeks to enter the lucrative intranasal mometasone furoate market that Nasonex® nasal spray has created with its beneficial and advantageous treatments for diseases of the upper airways, including allergic and nonallergic rhinitis.

COUNT I

20. Each of the preceding paragraphs is incorporated as if fully set forth herein.

21. On information and belief, Amneal filed ANDA No. 207989 to obtain approval under the FFDCA to engage in the commercial manufacture, use, or sale of a drug product which is claimed in the '353 patent, before the expiration of the '353 patent. On

information and belief, Amneal has committed an act of infringement under 35 U.S.C. § 271 (e)(2)(A), and Amneal will further infringe at least one claim of the '353 patent by making, using, offering to sell, and selling its generic copies of Nasonex® nasal spray in the United States and/or importing such copies into the United States unless enjoined by the Court.

22. On information and belief, when Amneal filed ANDA No. 207989 seeking approval to market generic mometasone furoate nasal spray before the expiration of the '353 patent, Amneal was aware of the existence of the '353 patent and that the filing of ANDA No. 207989 constituted an act of infringement of that patent.

23. On information and belief, Amneal acted without a reasonable basis for a good faith belief that it would not be liable for infringing the '353 patent.

24. If Amneal's marketing and sale of generic mometasone furoate nasal spray prior to expiration of the '353 patent and all other relevant exclusivities is not enjoined, Merck will suffer substantial and irreparable harm for which there is no remedy at law.

COUNT II

25. Each of the preceding paragraphs is incorporated as if fully set forth herein.

26. On information and belief, Amneal filed ANDA No. 207989 to obtain approval under the FFDCA to engage in the commercial manufacture, use, or sale of a drug product which is claimed in the '353 patent, before the expiration of the '353 patent. On information and belief, Amneal has committed an act of infringement under 35 U.S.C. § 271 (e)(2)(A), and Amneal will further contribute to the infringement of others of at least one claim of the '353 patent.

27. In any event, to the extent that Amneal contends that it will not directly infringe the ’353 patent by the manufacture, use, or sale of the product described in ANDA No. 207989, the mometasone furoate in Amneal’s product constitutes a material part of the invention of the ’353 patent because the mometasone furoate will be present in Amneal’s product in such a form and under such conditions so as to infringe the ’353 patent during storage during the proposed shelf life of such product and at the time of the use of such product.

28. Amneal has knowledge of the ’353 patent, as evidenced by at least its identification of the ’353 patent in connection with its filing of ANDA No. 207989.

29. On information and belief, Amneal has or will have knowledge that if it were to receive approval from the FDA to market the product described in ANDA No. 207989 and made said product available for sale and/or use during the proposed shelf life of the product, such activities would result in the sale and/or use of an infringing article that is not a staple article or commodity of commerce suitable for substantial noninfringing use, but rather is especially made and/or adapted for use in the direct infringement of the ’353 patent by another.

30. On information and belief, Amneal acted without a reasonable basis for a good faith belief that it would not be liable for contributing to the infringement the ’353 patent.

31. If Amneal’s marketing and sale of generic mometasone furoate nasal spray prior to expiration of the ’353 patent and all other relevant exclusivities is not enjoined, Merck will suffer substantial and irreparable harm for which there is no remedy at law.

REQUESTED RELIEF

WHEREFORE, Plaintiff Merck respectfully seeks the following relief:

a) That judgment be entered that Defendant Amneal has directly infringed and/or contributed to the infringement of the ’353 patent by submitting ANDA No. 207989;

b) That a permanent injunction be issued under 35 U.S.C. § 271(e) restraining or enjoining Defendant Amneal, its officers, agents or attorneys or employees, and those acting in privity or concert with them, from engaging in the commercial manufacture, use, offer to sell, or sale within the United States, or importation into the United States, of any chemical entity and/or therapeutic composition, covered by the '353 patent for the full term thereof, including the applicable pediatric exclusivity, and from contributing to such activities;

c) That an order be issued under 35 U.S.C. § 271(e)(4)(A) that the effective date of any approval of ANDA No. 207989 be a date which is not earlier than the expiration date of the asserted patent, including the applicable pediatric exclusivity;

d) That this is an exceptional case under 35 U.S.C.§ 285 and that judgment be entered for costs and reasonable attorney fees to be awarded to Merck; and

e) That this Court award such other and further relief as the Court may deem proper and just under the circumstances.

MORRIS, NICHOLS, ARSHT & TUNNELL LLP

/s/ Derek J. Fahnestock

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September 1, 2015
9424795

EXHIBIT 1

US006127353A

United States Patent [19]

Yuen et al.

[11] Patent Number: 6,127,353

[45] Date of Patent: Oct. 3, 2000

[54] **MOMETASONE FUROATE MONOHYDRATE, PROCESS FOR MAKING SAME AND PHARMACEUTICAL COMPOSITIONS**

[75] Inventors: **Pui-Ho Yuen**, Princeton Junction; **Charles Eckhart**, Kenilworth; **Teresa Etlinger**, Bloomfield; **Nancy Levine**, Flemington, all of N.J.

[73] Assignee: **Schering Corporation**, Kenilworth, N.J.

[21] Appl. No.: **07/984,573**

[22] PCT Filed: **Sep. 6, 1991**

[86] PCT No.: **PCT/US91/06249**

§ 371 Date: **Apr. 29, 1998**

§ 102(e) Date: **Apr. 29, 1998**

[51] **Int. Cl.**[7] **A61K 31/58**; C07J 17/00

[52] **U.S. Cl.** **514/172**; 514/171; 540/114

[58] **Field of Search** 540/115, 114; 514/172, 171

[56] **References Cited**

U.S. PATENT DOCUMENTS

4,472,393	9/1984	Shapiro	424/243
4,775,529	10/1988	Sequeira et al.	514/171
4,783,444	11/1988	Watkins et al.	514/19

FOREIGN PATENT DOCUMENTS

0262681	4/1988	European Pat. Off. .

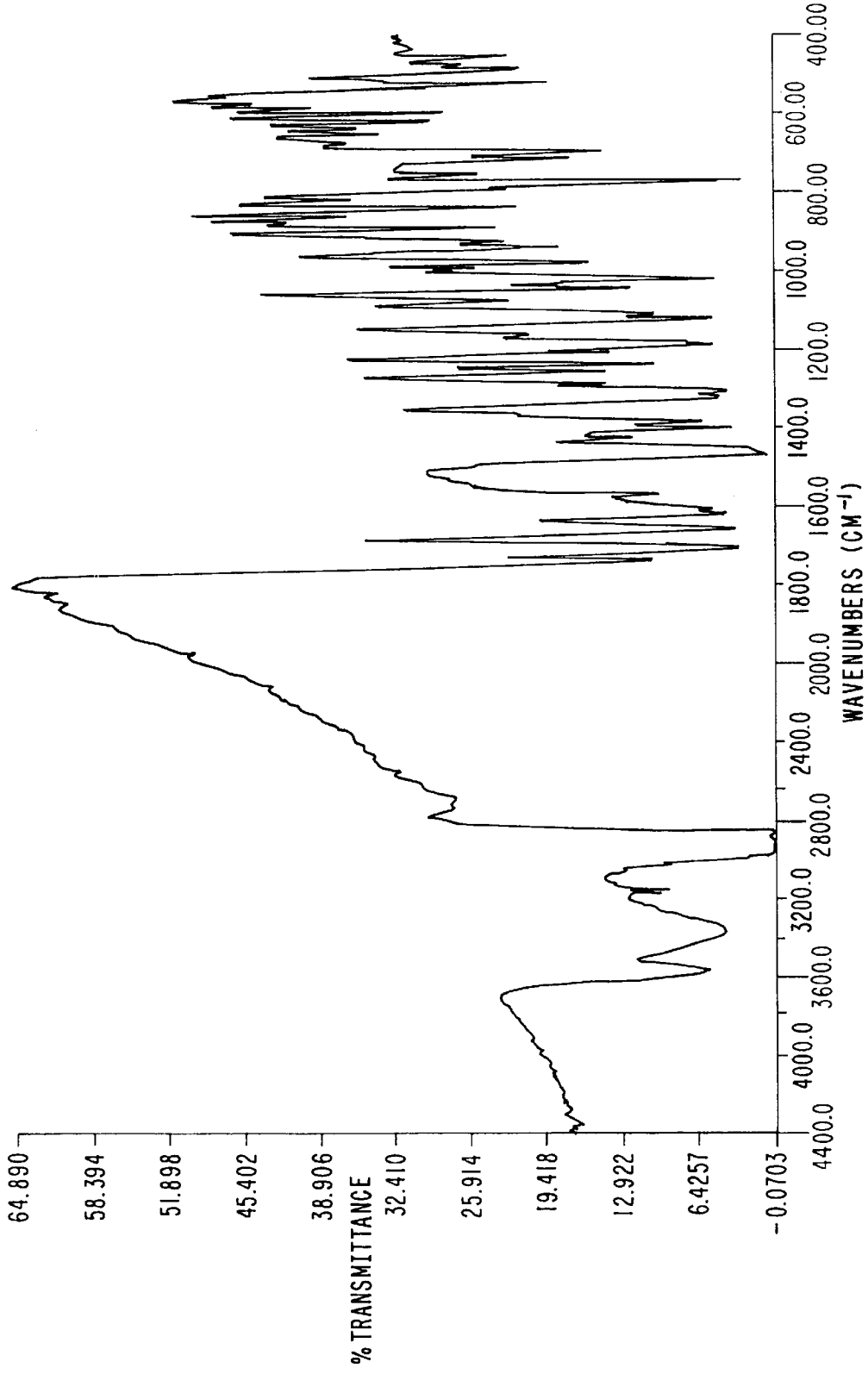
Primary Examiner—Jose'G. Dees
Assistant Examiner—Barbara Badio
Attorney, Agent, or Firm—John J. Maitner; Carl W. Battle; Robert A. Franks

[57] **ABSTRACT**

The invention relates to the novel compound mometasone furoate monohydrate, process for its preparation and pharmaceutical compositions containing said compound.

12 Claims, 2 Drawing Sheets

FIG. 1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58.394
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32.410
25.914
19.418
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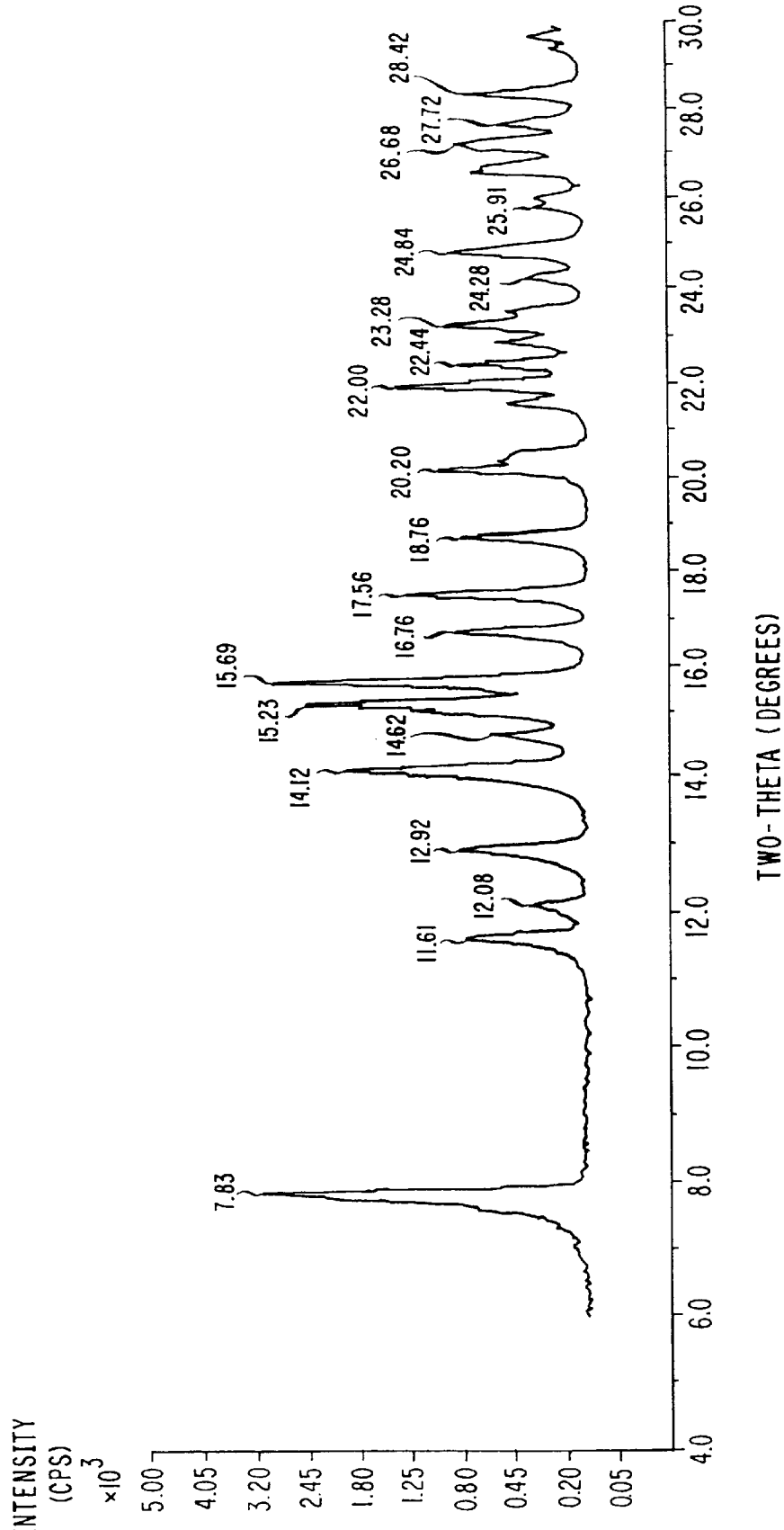
% TRANSMITTANCE
4400.0
4000.0
3600.0
3200.0
2800.0
2400.0
2000.0
1800.0
1600.0
1400.0
1200.0
1000.0
800.00
600.00
400.00
WAVENUMBERS (CM-1)

FIG. 2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5.00
4.05
3.20
2.45
1.80
1.25
0.80
0.45
0.20
0.05
4.0
6.0
8.0
10.0
12.0
14.0
16.0
18.0
20.0
22.0
24.0
26.0
28.0
30.0
TWO-THETA (DEGREES)
7.83
11.61
12.08
12.92
14.12
14.62
15.23
15.69
16.76
17.56
18.76
20.20
22.00
22.44
23.28
24.28
24.84
25.91
26.68
27.72
28.42

MOMETASONE FUROATE MONOHYDRATE, PROCESS FOR MAKING SAME AND PHARMACEUTICAL COMPOSITIONS

This application is a 371 of PCT/US91/06249 filed Sep. 6, 1991.

BACKGROUND OF THE INVENTION

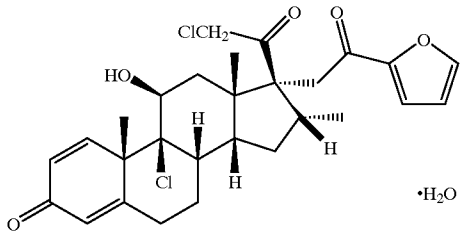
The present invention relates to a novel composition of matter, 9α, 21-dichloro-16α-methyl-1,4-pregnadiene-11β, 17α-diol-3,20-dione-17-(2'-furoate) monohydrate, also designated mometasone furoate monohydrate, process for its preparation, and pharmaceutical preparation thereof.

Mometasone furoate is known to be useful in the treatment of inflammatory conditions. The compound is prepared by procedures disclosed in U.S. Pat. No. 4,472,393, which patent is hereby incorporated by reference.

When aqueous pharmaceutical compositions, e.g. suspensions, containing anhydrous mometasone furoate were subjected to stability testing by rotating for four weeks at room temperature and 35° C., formation of a crystalline material which is different from the anhydrous mometasone furoate crystal was observed in suspension. Experiments were designed to determine the nature of the crystalline material. It was postulated that formulation of mometasone furoate compositions with the stable crystalline form would reduce the probability of crystal growth during long term storage of the suspension leading to a more stable product.

SUMMARY OF THE INVENTION

The present invention provides mometasone furoate monohydrate of formula I



a process for preparing said compound by crystallization from a saturated aqueous water miscible organic solution. The present invention also provides aqueous stable pharmaceutical compositions of mometasone furoate monohydrate.

DESCRIPTION OF THE FIGURES

FIG. **1**: Infrared spectrum of crystalline mometasone furoate monohydrate

FIG. **2**: X-ray diffraction pattern of crystalline mometasone furoate monohydrate

DETAILED DESCRIPTION OF THE INVENTION

The composition of matter of the present invention, mometasone furoate monohydrate has the following characteristics.

Molecular formula $C_{27}H_{30}Cl_2O_6H_2O$

Formula weight 539.46

Elemental Analysis (theory) C=60.11%, H=5.98%; Cl=13.16% (found) C=59.99%; H=5.56%; Cl=13.17%

Water Analysis (% H_2O) (theory) 3.34% (found) 3.31, 3.47

The crystalline mometasone furoate monohydrate exhibits an x-ray crystallographic powder diffraction pattern having essentially the values as shown in Table I.

TABLE 1

Angle of 2θ (degrees)	Spacing d (Å)	Relative Intensity I/I
7.795	11.3324	100
11.595	7.6256	6
12.035	7.3478	3
12.925	6.8437	11
14.070	6.2893	22
14.580	6.0704	5
14.985	5.9072	12
15.225	5.8146	33
15.635	5.6631	96
16.710	5.3011	15
17.515	5.0592	14
18.735	4.7324	12
20.175	4.3978	13
20.355	4.3593	6
20.520	4.3246	4
21.600	4.1108	5
21.985	4.0396	22
22.420	3.9622	8
22.895	3.8811	7
22.245	3.8234	14
23.550	3.7746	13
24.245	3.6680	4
24.795	3.5878	11
24.900	3.5729	5
24.800	3.4503	5
25.985	3.4262	3
26.775	3.3268	84
27.170	3.2794	10
27.305	3.2635	9
27.710	3.2167	5
28.385	3.1417	7
29.165	3.0594	1
29.425	3.0330	2
29.725	3.0030	2
30.095	2.9670	7
30.255	2.1516	3
30.490	2.9294	10
30.725	2.9075	6
31.115	2.8720	3
31.595	2.8294	47
32.135	2.7831	6
32.985	2.7133	7
33.400	2.6805	2
33.820	2.6482	2
34.060	2.6301	8
34.625	2.5885	4
34.795	2.5762	2
35.315	2.5394	1
36.780	2.4416	21
37.295	2.4090	2

Single crystal data of mometasone furoate monohydrate exhibits the following values as shown in Table II.

TABLE II

Crystallographic Data[a]	
Crystal system	triclinic
Space group	$P1(C^1_1)$-No. 1
a (Å)	8.481 (1)
b (Å)	11.816 (2)
c(Å)	7.323 (1)
α(°)	95.00 (1)
β(°)	110.66 (1)
γ(°)	73.27 (1)

TABLE II-continued

Crystallographic Data[a]	
Crystal system	triclinic
Space group	P1(C^1_1)-No. 1
V (Å^3)	657.5 (3)
$D_{calcd.}$ (g cm^{-3})	1.362

a An Enraf-Nonius CAD-4 diffractometer (Cu-Kα radiation, incident-beam graphite monochromator) was used for all measurements, Intensity data were corrected for the usual Lorentz and polarization effects; an empirical absorption correction was also applied.

The crystal structure was solved by direct methods (RANTAN). Approximate non-hydrogen atom positions were derived from an E-map. Hydrogen atoms were located in a series of difference Fourier syntheses evaluated following several rounds of full-matrix least-squares adjustment of non-hydrogen atom positional and anisotropic temperature factor parameters. Hydrogen atom positional and isotropic thermal parameters were included as variables in the later least-squares iterations which also involved refinement of an extinction correction. Crystallographic calculations were performed on PDP11/44 and MicroVAX computers by use of the Enfra-Nonius Structure Determination Package (SDP). For all structure-factor calculations, neutral atom scattering factors and their anomalous dispersion corrections were taken from *International Tables for X-Ray Crystallography,* vol. IV, The Knynock Press, Birmingham, England, 1974.

Mometasone furoate monohydrate can be prepared by forming a saturated homogeneous solution of anhydrous mometasone furoate in a mixture of water and a water miscible organic solvent. The saturated solution is prepared by dissolving the mometasone furoate in a water miscible organic solvent at the temperature of about 85° C. Hot water, about 85° C., is added dropwise with agitation. After removing the solution from the steam bath, the reaction is stirred for about one hour and then allowed to stand undisturbed overnight while cooling to room temperature. The solution is stirred while adding additional water at room temperature and the solution becomes cloudy and a white precipitate forms. The reaction is allowed to stir for a time, the preciptitate collected by filtration and the product dried to constant weight.

Organic solvents that can be employed in the process of this invention must be miscible with water and one in which mometasone furoate is soluble. Examples of water miscible organic solvents include alcohols, such as, ethanol, isopropanol, and the like; ketones, such as acetone, and the like; ethers, such as dioxane, and the like; esters such as ethyl acetate, and the like. The preferred solvents are acetone and isopropanol.

In another aspect, the present invention provides pharmaceutical compositions comprising mometasone furoate monohydrate of formula I in an inert pharmaceutically acceptable carrier or diluent.

The pharmaceutical compositions according to the invention can be prepared by combining mometasone furoate monohydrate with any suitable inert pharmaceutical carrier or diluent and administered orally, parentally or topically in a variety of formulations.

Of particular interest are aqueous suspension compositions of mometasone furoate monohydrate, e.g. for nasal administration. The aqueous suspensions of the invention may contain from 0.1 to 10.0 mg of mometasone furoate monohydrate per gram of suspension.

The aqueous suspension compositions according to the present invention may contain, inter alia, auxiliaries and/or more of the excipients, such as: suspending agents, e.g. microcrystalline cellulose, sodium carboxymethylcellulose, hydroxypropyl-methyl cellulose; humectants, e.g. glycerin and propylene glycol; acids, bases or buffer substances for adjusting the pH, e.g. citric acid, sodium citrate, phosphoric acid, sodium phosphate e.g. citrate and phosphate buffers; surfactants, e.g. Polysorbate 80; and antimicrobial preservatives, e.g. benzalkonium chloride, phenylethyl alcohol and potassium sorbate.

The following examples illustrate the present invention and the best made of practicing the process of the invention. It will be apparent to those skilled in the art that modifications thereof may be practical without departing from the purpose and intent of this disclosure.

General Experimental

Infrared absorption spectra were taken as Nujol Mull on a Nicolet FT-infrared spectrometer Model No. 5DXB. X-ray crystallograph powder diffraction patterns were taken on a Philips X-ray diffractometer Model APD-3720 equipped with a radiation source: copper Kα. Decomposition temperatures were measured on a Dupont differential scanning calorimeter, Model No. 990.

Moisture content of the crystalline mometasone furoate monohydrate was determined by titration with Karl Fisher reagent.

EXAMPLE 1

Place 4.5 liters of ethyl alcohol into a suitable vessel equipped with an appropriate agitator and closure. Dissolve 27 g of mometasone furoate anhydrous powder into the ethanol with stirring. Filter the saturated solution and slowly add purified water about 1.5 liters, at a flow rate of approximately 50 ml/minute while stirring at moderate speed. When the solvent mixture reaches a ratio of 1:3 (water:ethanol), the addition of water is stopped and stirring of the reaction mixture is continued for approximately 2 hours to facilitate seeding. Resume addition of water, about 7.5 liters at a rate of approximately 50 ml/minute, until a ratio of 2:1 (water:ethanol) is achieved. Continue stirring to complete crystallization. The crystals are collected by filtration and dried in a vacuum desiccator at room temperature to afford 24.83 g of mometasone furoate monohydrate having an infrared spectrum and X-ray diffraction graph substantially the same as that in FIGS. **1** and **2**.

EXAMPLE 2

Place 24.3 liters of 2-propanol into a suitable container. Dissolve 340 grams of anhydrous mometasone furoate in the 2-propanol by heating the mixture (steam bath) to 85° C. with stirring. After the furoate has dissolved, add dropwise with stirring over 15 minutes 1950 ml of hot (85° C.) water. The hot solution is removed from the steam bath and the solution is stirred for 1 hour. The solution is allowed to cool to room temperature overnight without stirring. The remainder of water, about 24 liters is added with stirring; the solution becomes cloudy and a white precipitate begins to form. The reaction is stirred for one hour, following addition of the water. The white precipitate is collected by filtration, washed with 2 liters of water and air dried overnight. The solid is dried in a draft oven at 50° C. to constant weight. Mometasone furoate monohydrate, 316.5 g, weight yield 90%, is obtained having an infrared spectrum and X-ray diffraction graph substantially the same as that in FIGS. **1** and **2**.

EXAMPLE 3

An aqueous nasal suspension of mometasone furoate monohydrate is prepared from the following:

Ingredients	Concentration mg/g	Representative Batch g/12 kg
Mometasone furoate monohydrate	0.5	6.0
Avicel RC 591*	20.0	240.0
Glycerin	21.0	252.0
Citric Acid	2.0	24.0
Sodium citrate	2.8	33.6
Polysorbate 80**	0.1	1.2
Benzalkonium chloride	0.2	2.4
Phenylethyl alcohol	2.5	30.0
Purified water q.s. ad	1.0 g	12.0 kg

*Avicel RC-591-is a trademark of FMC for a mixture of microcrystalline cellulose and sodium carboxymethyl cellulose.
**Polysorbate 80 is a tradename for a mixture of an oleate ester of sorbitol and its anhydride copolymerized with approximately 20 moles of ethylene oxide for each mole of sorbitol and sorbitol anhydride.

After dispersing the Avicel RC 591 in 6 kg of purified water, the glycerin is added thereto. The citric acid and sodium citrate is dissolved in 240 ml of water, said solution is added to the Avicel-glycerin dispersion with mixing. In a separate vessel, Polysorbate 80 is dissolved in approximately 400 ml of purified water with stirring. The mometasone furoate monohydrate is dispersed in the aqueous Polysorbate 80 solution and; said slurry is then added with stirring to the Avicel-glycerin citric acid mixture. After dissolving benzalkonuim chloride and phenylethyl alcohol in purified water, said solution is added to the suspension mixture with stirring. The suspension is brought to 12 kg with purified water with mixing. The final pH of the suspension is 4.5±0.5.

EXAMPLE 4

The following compositions were prepared without the suspending agent, Avicel RC-591 to prevent interference in X-ray diffraction studies:

	Concentration mg/g		
Ingredients	4A	4B	4C
Mometasone Furoate Monohydrate Micronized	0.5	0.5	0.5
Citric Acid Monohydrate	2.0	2.0	2.0
Sodium Citrate Dihydrate	2.8	—	2.8
Sodium Phosphate Dibasic	—	4.0	—
Polysorbate 80	0.1	0.1	0.1
Benzalkonium Chloride	0.2	0.2	0.2
Phenylethyl Alcohol	2.5	—	—
Potassium Sorbate	—	3.4	—
Propylene Glycol	—	—	100.0
Glycerin	21.0	21.0	21.0
Water Purified USP q.s. ad	1.0 g	1.0 g	1.0 g

These compositions were prepared according to the procedure described in Example 3.

The three compositions 4A, 4B and 4C were rotated for five (5) days at 35° C. and a additional four (4) weeks at room temperature to assess crystal form stability. The crystals were isolated from the suspension and X-ray diffraction patterns determined. The results indicated that the crystals collected from each of the three compositions are in the form of mometasone furoate monohydrate.

EXAMPLE 5

The following compositions were prepared and tested to determine thermal stability of said compositions.

	Concentration mg/g		
Ingredients	4A	4B	4C
Mometasone Furoate Monohydrate Micronized	0.5	0.5	0.5
Citric Acid Monohydrate	2.0	2.0	2.0
Sodium Citrate Dihydrate	2.8	—	2.8
Sodium Phosphate Dibasic	—	4.0	—
Polysorbate 80	0.1	0.1	0.1
Benzalkonium Chloride	0.2	0.2	0.2
Phenylethyl Alcohol	—	2.5	—
Potassium Sorbate	—	—	3.4
Propylene Glycol	100.0	—	—
Glycerin	21.0	21.0	21.0
Avicel RC-591	20.0	20.0	20.0
Water Purified USP q.s. ad	1.0 g	1.0 g	1.0 g

The compositions were prepared according to the procedure described in Example 3.

The compositions were thermally cycled between 4° C. (24 hours) and 30° C. (24 hours) for a period of one month. Microscopic analyses revealed no detectable mometasone furoate monohydrate crystal growth under these conditions.

We claim:

1. 9α,21-dichloro-16α-methyl-1,4-pregnadiene-11β,17α-diol-3,20-dione-17-(2'-furoate) monohydrate.

2. A pharmaceutical composition comprising an antiinflammatory amount of mometasone furoate monohydrate in a pharmaceutically acceptable carrier.

3. The composition of claim **2**, further comprising the following ingredients:

Ingredients	mg/g
Mometasone furoate monohydrate	0.1–10
Microcrystalline cellulose and sodium carboxymethyl cellulose	20
Glycerin	21
Citric acid	2
Sodium citrate	2.8
Polysorbate 80	0.1
Benzalkonium chloride	0.2
Phenylethyl alcohol	2.5
Purified water q.s. ad	1.0 g

4. The composition of claim **3** comprising 0.5 mg of mometasone furoate monohydrate.

5. The compound 9α,21-dichloro-16α-methyl-1,4-pregnadiene-11β,17α-diol-3,20 dione-17-(2'-furoate) monohydrate exhibiting a x-ray crystallographic powder diffraction pattern having essentially the following values:

Angle of 2θ (degrees)	Spacing d (Å)	Relative Intensity I/I
7.795	11.3324	100
11.595	7.6256	6
12.035	7.3478	3
12.925	6.8437	11

-continued

Angle of 2θ (degrees)	Spacing d (Å)	Relative Intensity I/I
14.070	6.2893	22
14.580	6.0704	5
14.985	5.9072	12
15.225	5.8146	33
15.635	5.6631	96
16.710	5.3011	15
17.515	5.0592	14
18.735	4.7324	12
20.175	4.3978	13
20.355	4.3593	6
20.520	4.3246	4
21.600	3.1108	5
21.985	3.0396	22
22.420	3.9622	8
22.895	3.8811	7
23.245	3.8234	14
23.550	3.7746	13
24.245	3.6680	4
24.795	3.5878	11
24.900	3.5729	5
25.800	3.4503	5
25.985	3.4262	3
26.775	3.3268	84
27.170	3.2794	10
27.305	3.2635	9
27.710	3.2167	5
28.385	3.1417	7
29.165	3.0594	1
29.425	3.0330	2
29.725	3.0030	2
30.095	3.9670	7
30.255	2.9516	3
30.490	2.9294	10
30.725	2.9075	6
31.115	2.8720	3
31.595	2.8294	47
32.135	2.7831	6
32.985	2.7133	7
33.400	2.6805	2

-continued

Angle of 2θ (degrees)	Spacing d (Å)	Relative Intensity I/I
33.820	2.6482	2
34.060	2.6301	8
34.625	2.5885	4
34.795	2.5762	2
35.315	2.5394	1
36.780	2.4416	21
37.295	2.4090	2

6. A pharmaceutical composition comprising mometasone furoate monohydrate in a carrier consisting essentially of water.

7. The pharmaceutical composition of claim **6** wherein said mometasone furoate monohydrate is present in an amount of from about 0.1 mg. to about 10.0 mg per gram of water.

8. The pharmaceutical composition of claim **6** wherein said mometasone furoate monohydrate is present in the form of micronized particles.

9. The pharmaceutical composition of claim **6** wherein said composition has a pH of from about 4.0 to 5.0.

10. The pharmaceutical composition of claim **6** further comprising one or more additional components selected from the group consisting essentially of excipients, suspending agents, buffers, surfactants, preservatives and mixtures thereof.

11. The pharmaceutical composition of claim **6** formulated as a nasal spray.

12. The pharmaceutical composition of claim **6** wherein said mometasone furoate monohydrate is suspended in said aqueous carrier.

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