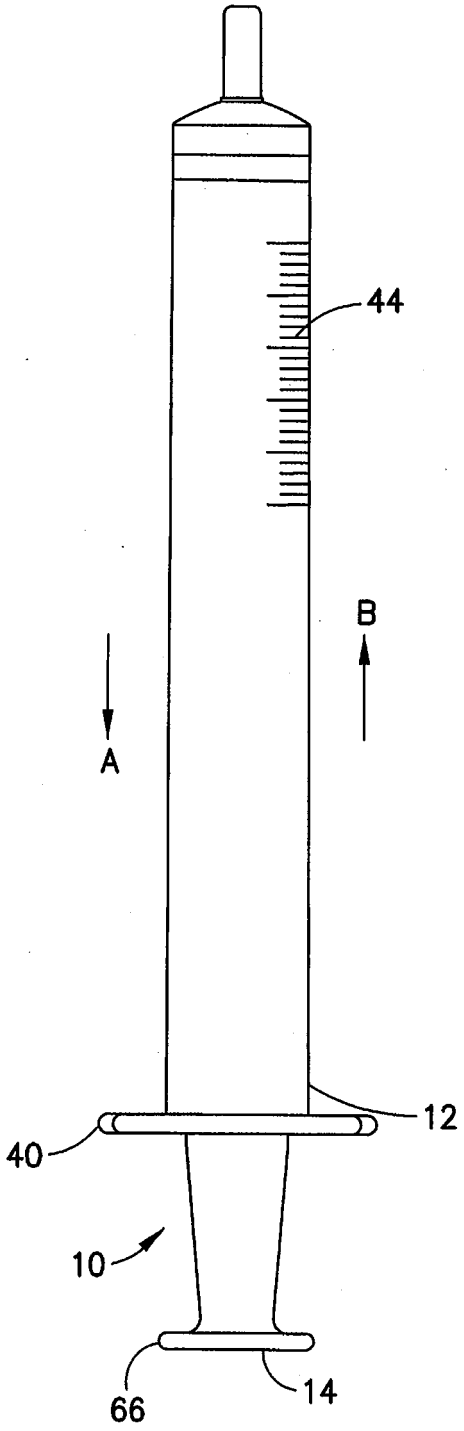


FIG.1

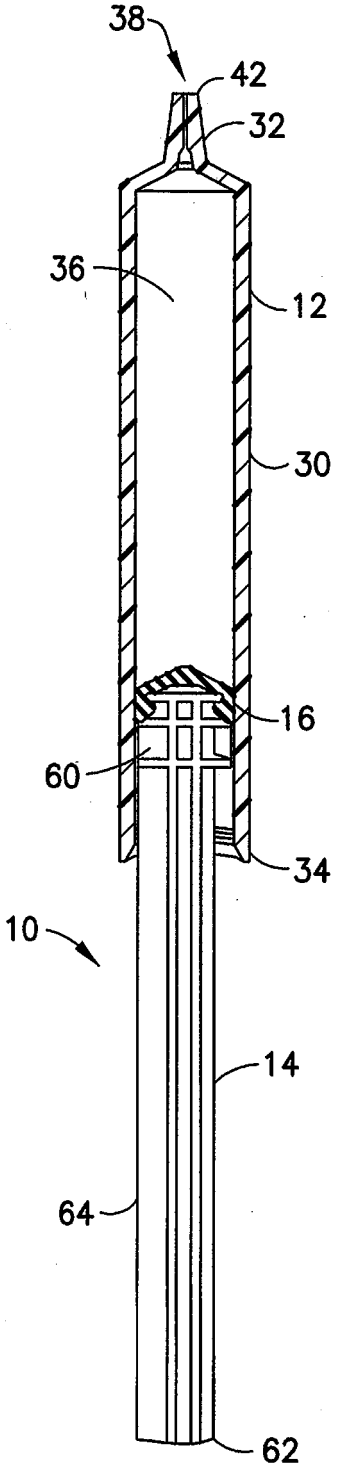


FIG.2A

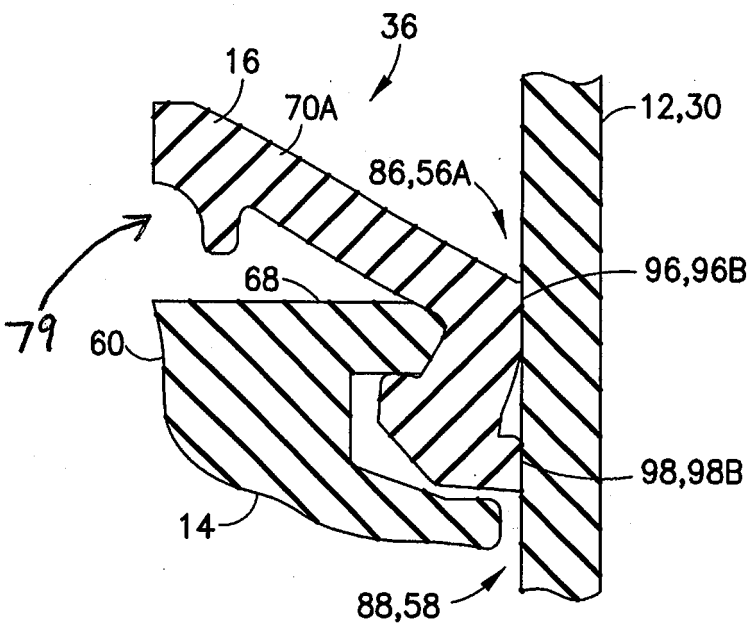

36
16
70A
12,30
86,56A
96,96B
68
79
60
98,98B
14
88,58

FIG.2B

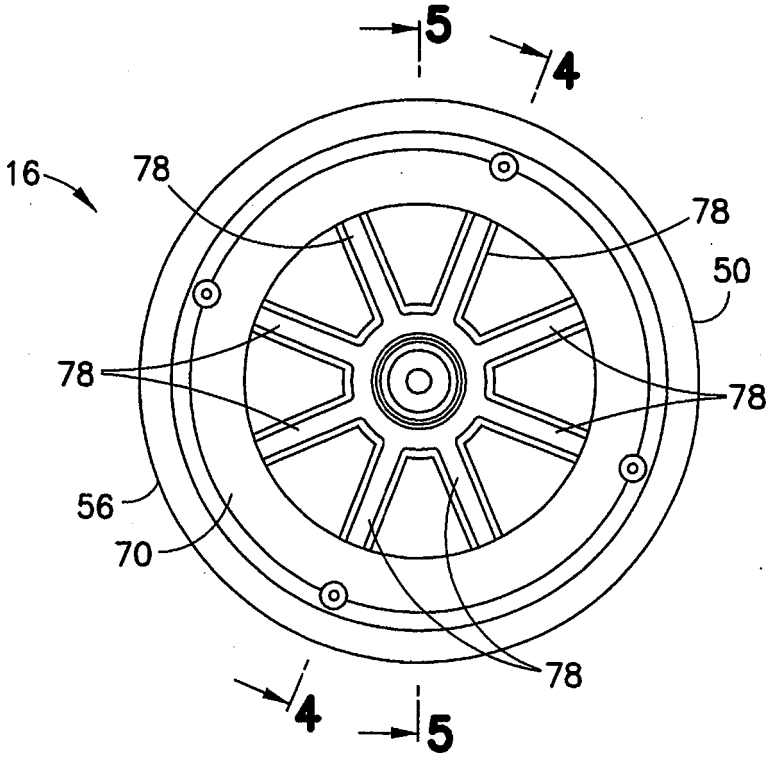

5
4
16
78
78
50
78
78
56
70
78
4
5

FIG.3

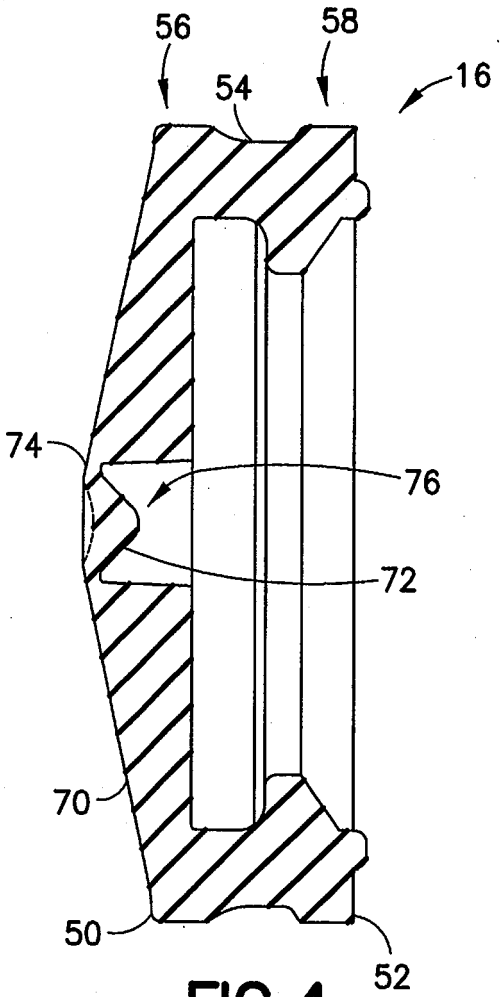


FIG.4

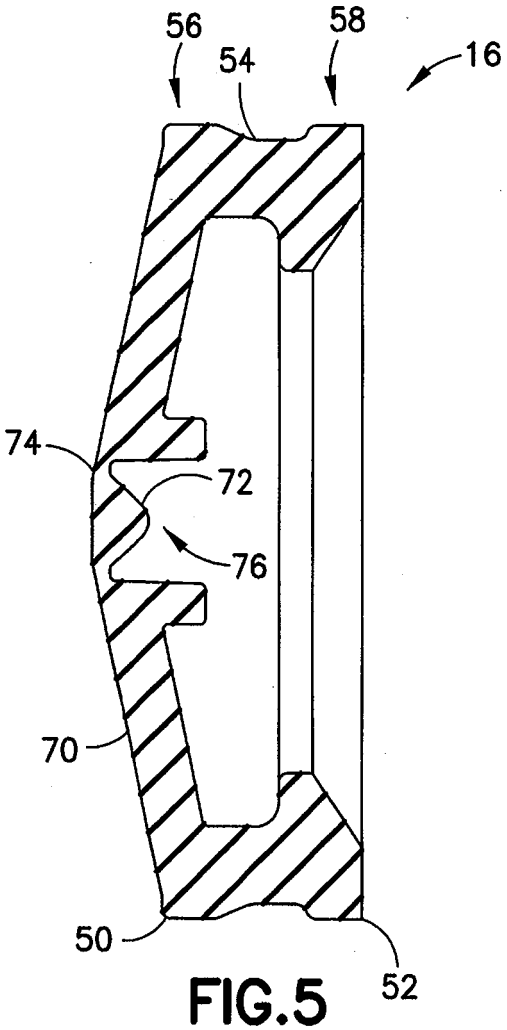


FIG.5

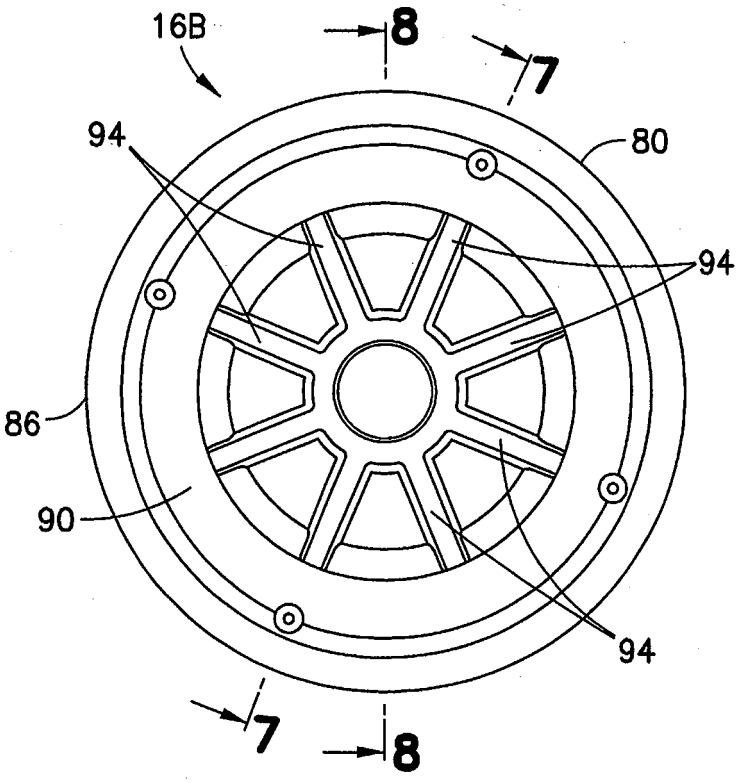


FIG.6

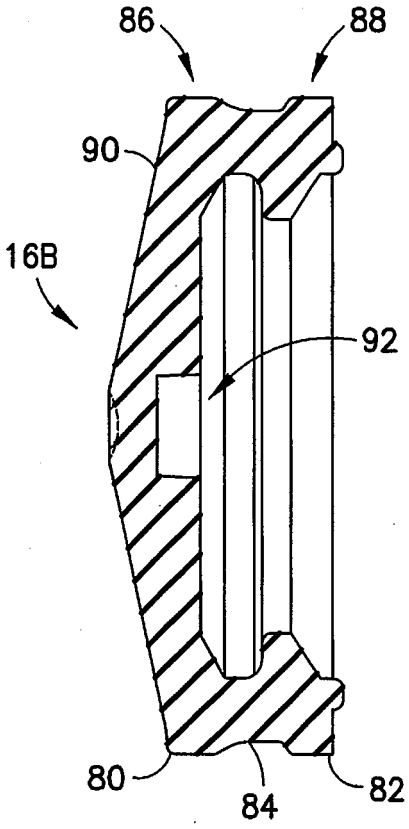


FIG.7

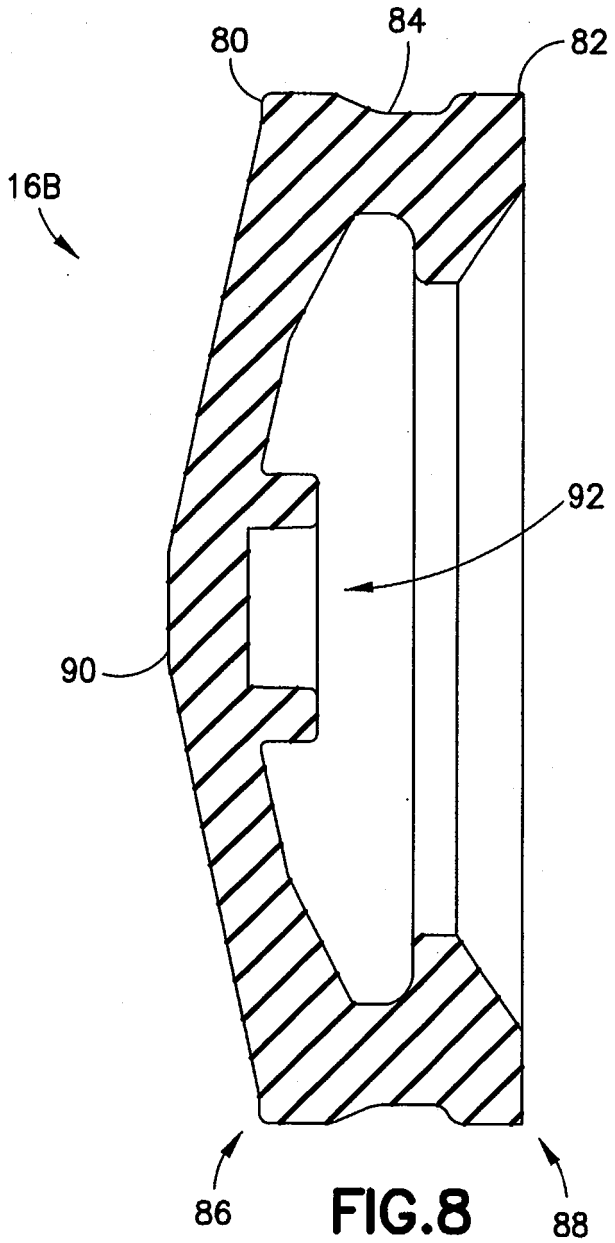


FIG.8

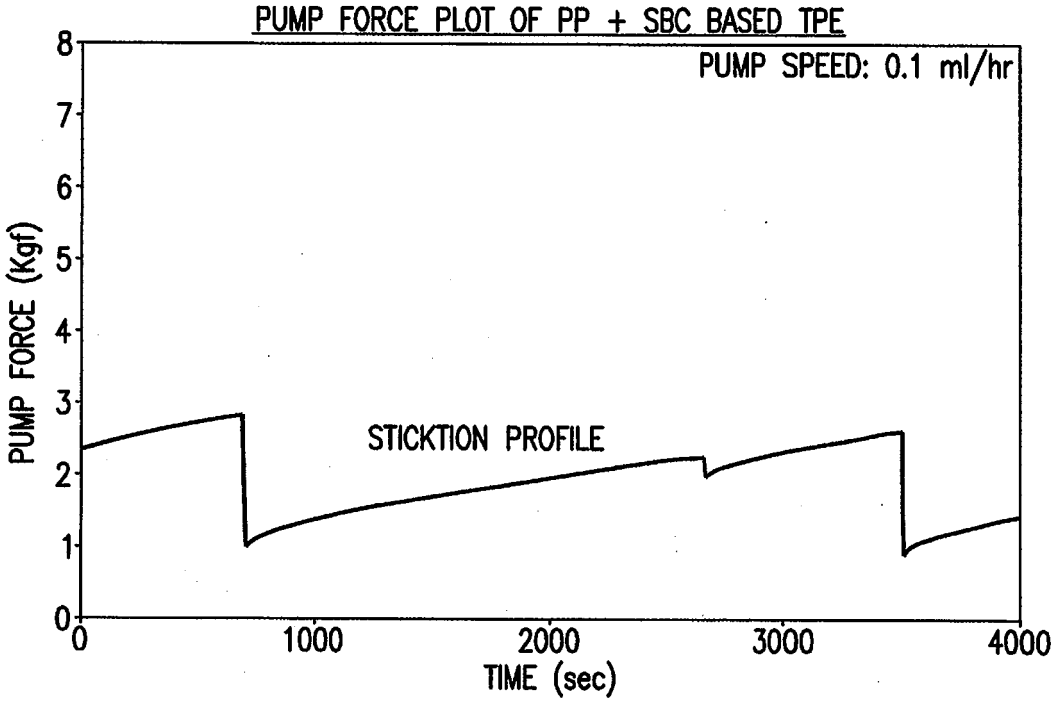

PUMP FORCE PLOT OF PP + SBC BASED TPE
PUMP SPEED: 0.1 ml/hr
PUMP FORCE (Kgf)
STICKTION PROFILE
TIME (sec)
0
1
2
3
4
5
6
7
8
0
1000
2000
3000
4000

FIG.9

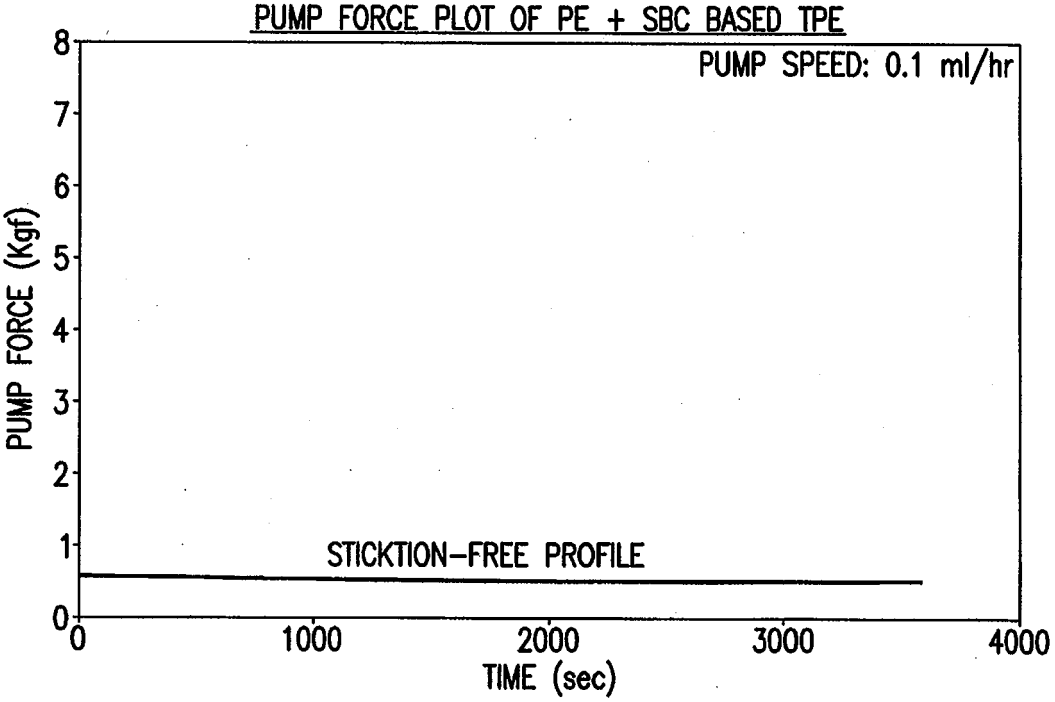

PUMP FORCE PLOT OF PE + SBC BASED TPE
PUMP SPEED: 0.1 ml/hr
PUMP FORCE (Kgf)
STICKTION-FREE PROFILE
TIME (sec)
0
1
2
3
4
5
6
7
8
0
1000
2000
3000
4000

FIG.10

STRAIN	DESIRED STRESS (psi)	TPE-1 STRESS (psi)	TPE-2 STRESS (psi)	TPE-3 STRESS (psi)
-25%	-143	-809	-1147	-314
-10%	-50	-170	-235	-80
10%	45	85	110	48
25%	94	142	90	91

FIG.11

TPE/RUBBER IN 10 ml, GAMMA STERILE. T=0	LEAK PRESSURE (psi), AVG/ST DEV, IT1499-01	BREAK-LOOSE FORCE (lbf), AVG/ST DEV, IT16-01	BREAK-OUT FORCE (lbf), AVG/ST DEV, IT16-01	SUSTAINING FORCE (lbf), AVG/ST DEV, IT16-01
CONVENTIONAL RUBBER DESIGN 1	189.0/29.9	3.9/0.6	1.2/0.2	0.9/0.1
TPE-1 STOPPER, DESIGN 2	102.9/10.3	5.8/0.6	1.5/0.2	1.0/0.1
TPE-1 STOPPER, DESIGN 3	204.0/14.3	3.5/0.5	0.9/0.1	0.5/0.1

FIG.12

TPE, GAMMA STERILE SYRINGE, 10ml DESIGN-4, T=0	BREAK-LOOSE FORCE (lbf), AVG/ST DEV, IT16-01	BREAK-OUT FORCE (lbf), AVG/ST DEV, IT16-01	SUSTAINING FORCE (lbf), AVG/ST DEV, IT16-01
TPE-1-S0.6, LUBED STOPPER	3.5/0.5	0.9/0.1	0.5/0.1
TPE-1-S0.6, UNLUBED STOPPER	3.3/0.7	0.9/0.1	0.5/0.1
TPE-2-S0.6, LUBED STOPPER	3.2/0.5	0.8/0.1	0.8/0.1
TPE-2-S0.6, UNLUBED STOPPER	3.8/0.4	0.7/0.1	0.5/0.1

FIG.13

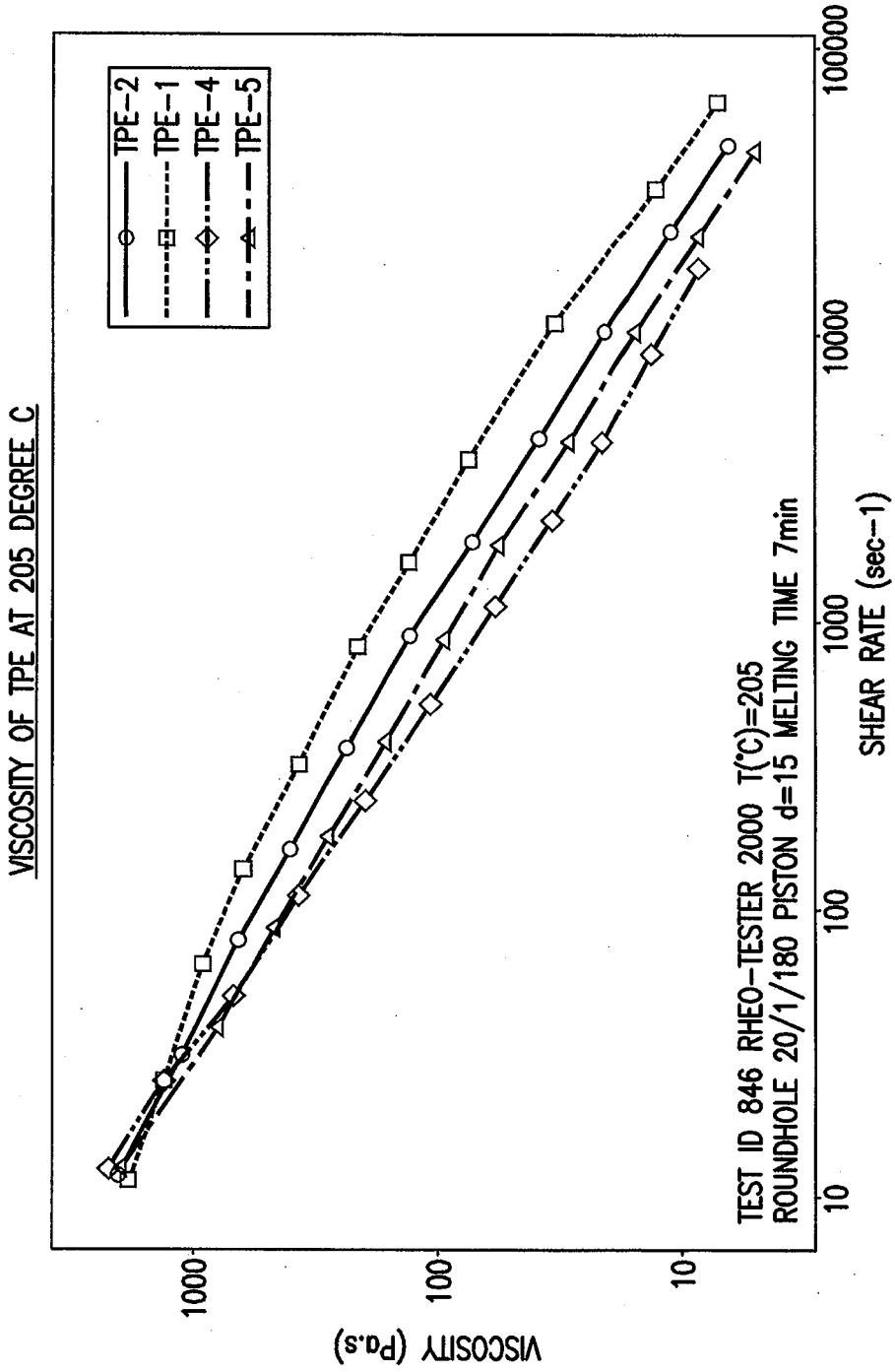

VISCOSITY OF TPE AT 205 DEGREE C
TPE-2
TPE-1
TPE-4
TPE-5
TEST ID 846 RHEO-TESTER 2000 T(°C)=205
ROUNDHOLE 20/1/180 PISTON d=15 MELTING TIME 7min
VISCOSITY (Pa.s)
1000
100
10
10
100
1000
10000
100000
SHEAR RATE (sec-1)

FIG.14A

TPE STOPPER IN DESIGN-5, 10ml SYRINGE, UNLUBED STOPPER, NON-STERILE SYRINGE, NO NEEDLE ATTACHED	ISOPROPANOL HAND CONTROL	SHEEP BLOOD HAND CONTROL
TPE-1	GOOD CONTROL AT DROPLET LEVEL	GOOD CONTROL AT DROPLET LEVEL
TPE-2	GOOD CONTROL AT DROPLET LEVEL	GOOD CONTROL AT DROPLET LEVEL
TPE-4	BAD CONTROL WITH SPURTING	BAD CONTROL WITH SPURTING
TPE-5	BAD CONTROL WITH SPURTING	BAD CONTROL WITH SPURTING

FIG.14B

10ml SYRINGE, NON-STERILE	FLUID PRESSURE (psi)	CONTACT PRESSURE USING FINITE ELEMENT ANALYSIS (psi)	NORMALIZED CONTACT PRESSURE USING FINITE ELEMENT ANALYSIS (psi)
STOPPER DESIGN-1, CONVENTIONAL RUBBER-2	0	104	1
	67	139	1.34
	133	38	0.37
STOPPER DESIGN-3, TPE-1	0	206	1
	67	383	1.86
	133	435	2.11

FIG.15

TPE STOPPER CANDIDATE	DESCRIPTION
TPE-1-S0.6	OLEFIN BLOCK COPOLYMER BASED WITH SMALL AMOUNT OF POLYPROPYLENE AS COLORANT CARRIER. CONTAINS 0.6% OF ERUCAMIDE SLIP AGENT
TPE-2-S0	POLYETHYLENE BLENDED WITH STYRENIC BLOCK COPOLYMER BASED WITH SMALL AMOUNT OF POLYPROPYLENE AS COLORANT CARRIER. CONTAINS DIFFERENT LEVEL OF ERUCAMIDE SLIP AGENT (0%, 0.1%, 0.2%, 0.3%, AND 0.6%)
TPE-2-S0.1	
TPE-2-S0.2	
TPE-2-S0.3	
TPE-2-S0.6	
TPE-3	POLYETHYLENE BLENDED WITH STYRENIC BLOCK COPOLYMER BASED WITH SMALL AMOUNT OF POLYPROPYLENE AS COLORANT CARRIER AND HAVING A SECOND COMPOSITION
TPE-4	POLYPROPYLENE BLENDED WITH STYRENIC BLOCK COPOLYMER BASED AND OF LOWER VISCOSITY THAN TPE-1 AND TPE-2
TPE-5	POLYETHYLENE BLENDED WITH STYRENIC BLOCK COPOLYMER BASED
TPE-6	POLYETHYLENE BLENDED WITH STYRENIC BLOCK COPOLYMER BASED HAVING A SECOND COMPOSITION
TPE-7	OLEFIN BLOCK COPOLYMER FROM DOW (GRADE: 9807)
TPE-8	OLEFIN BLOCK COPOLYMER FROM DOW (GRADE: 9507)
TPE-9	OLEFIN BLOCK COPOLYMER FROM DOW (GRADE: 9817)
TPE-10	POLYETHYLENE BLENDED WITH EPDM TPV PRODUCED WITH POLYPROPYLENE AS CONTINUOUS PHASE
TPE-11	POLYETHYLENE BLENDED WITH EPDM TPV PRODUCED WITH POLYPROPYLENE AS CONTINUOUS PHASE AND HAVING A SECOND COMPOSITION
TPE-12	POLYETHYLENE BLENDED WITH EPDM TPV PRODUCED WITH POLYPROPYLENE AS CONTINUOUS PHASE AND HAVING A THIRD COMPOSITION
TPE-13	POLYPROPYLENE AND POLYETHYLENE BLENDED WITH STYRENIC BLOCK COPOLYMER
TPE-14	POLYPROPYLENE BLENDED WITH STYRENIC BLOCK COPOLYMER BASED HAVING A SECOND COMPOSITION
TPE-15	POLYPROPYLENE BLENDED WITH STYRENIC BLOCK COPOLYMER BASED HAVING A THIRD COMPOSITION

FIG.16

TPE STOPPER MATERIAL	PP CONTENT (%)	STICK-SLIP PERFORMANCE (PASS/FAIL)
TPE-1	<1	PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-2-S0	<1	PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-2-S0.1		PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-2-S0.2		PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-2-S0.3		PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-2-S0.6		PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-3	<1	PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-4	20	FAIL (5/5 SYRINGES EXHIBIT STICK-SLIP)
TPE-5	<1	PASS
TPE-6	<1	FAIL (5/5 SYRINGES EXHIBIT STICK-SLIP)
TPE-7	N/A	PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-8	N/A	PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-9	N/A	PASS (0/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-10	1.03	FAIL (1/5 SYRINGE EXHIBIT STICK-SLIP)
TPE-11	1.14	FAIL (2/5 SYRINGES EXHIBIT STICK-SLIP)
TPE-12	1.19	FAIL (2/5 SYRINGES EXHIBIT STICK-SLIP)
TPE-13	1.90	FAIL (3/5 SYRINGES EXHIBIT STICK-SLIP)
TPE-14	5.66	FAIL (3/3 SYRINGES EXHIBIT STICK-SLIP)
TPE-15	7.91	FAIL (5/5 SYRINGES EXHIBIT STICK-SLIP)

FIG.17

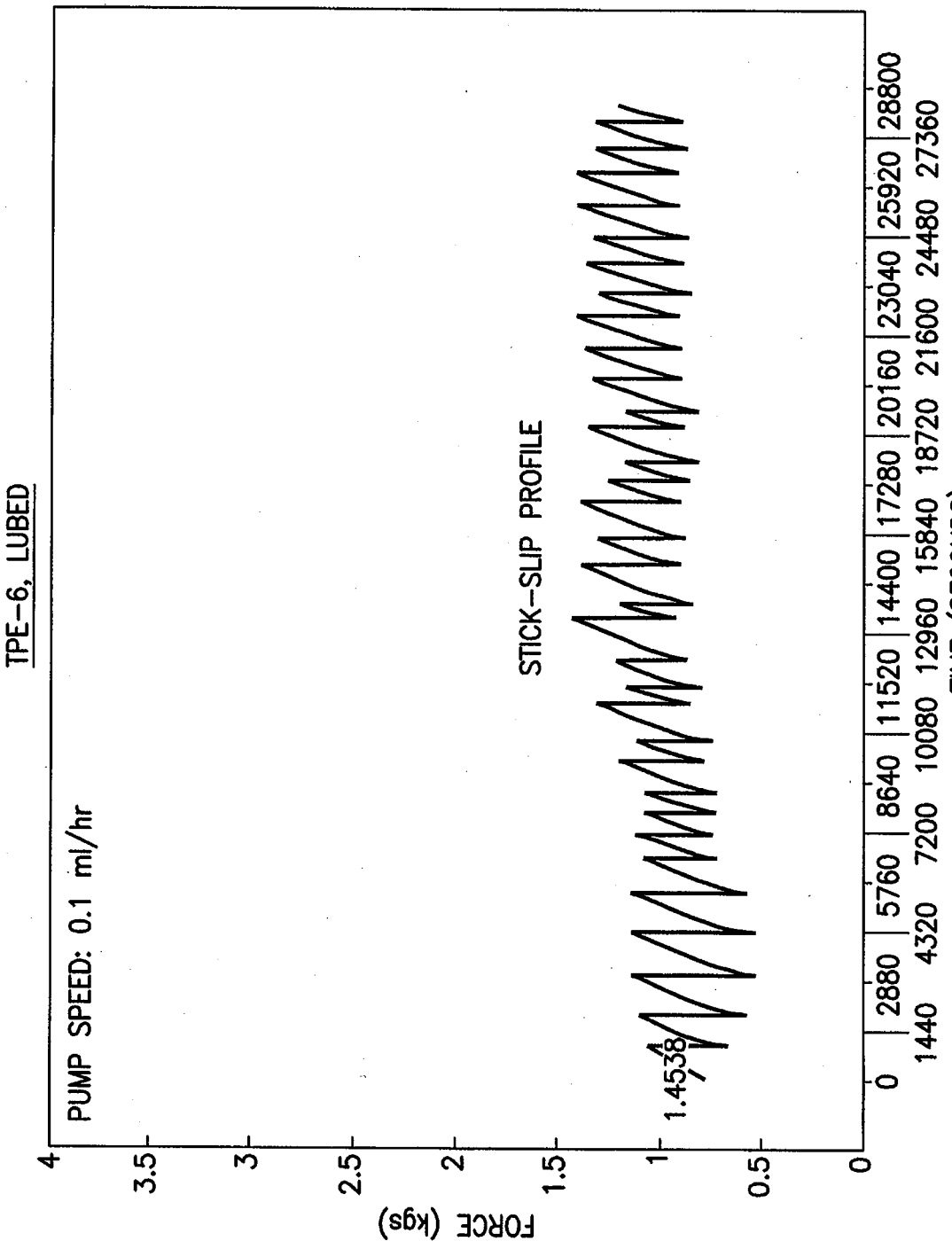

TPE-6, LUBED
PUMP SPEED: 0.1 ml/hr
STICK-SLIP PROFILE
FORCE (kgs)
0
0.5
1
1.5
2
2.5
3
3.5
4
1.4538
TIME (SECONDS)
0
1440
2880
4320
5760
7200
8640
10080
11520
12960
14400
15840
17280
18720
20160
21600
23040
24480
25920
27360
28800

FIG.18

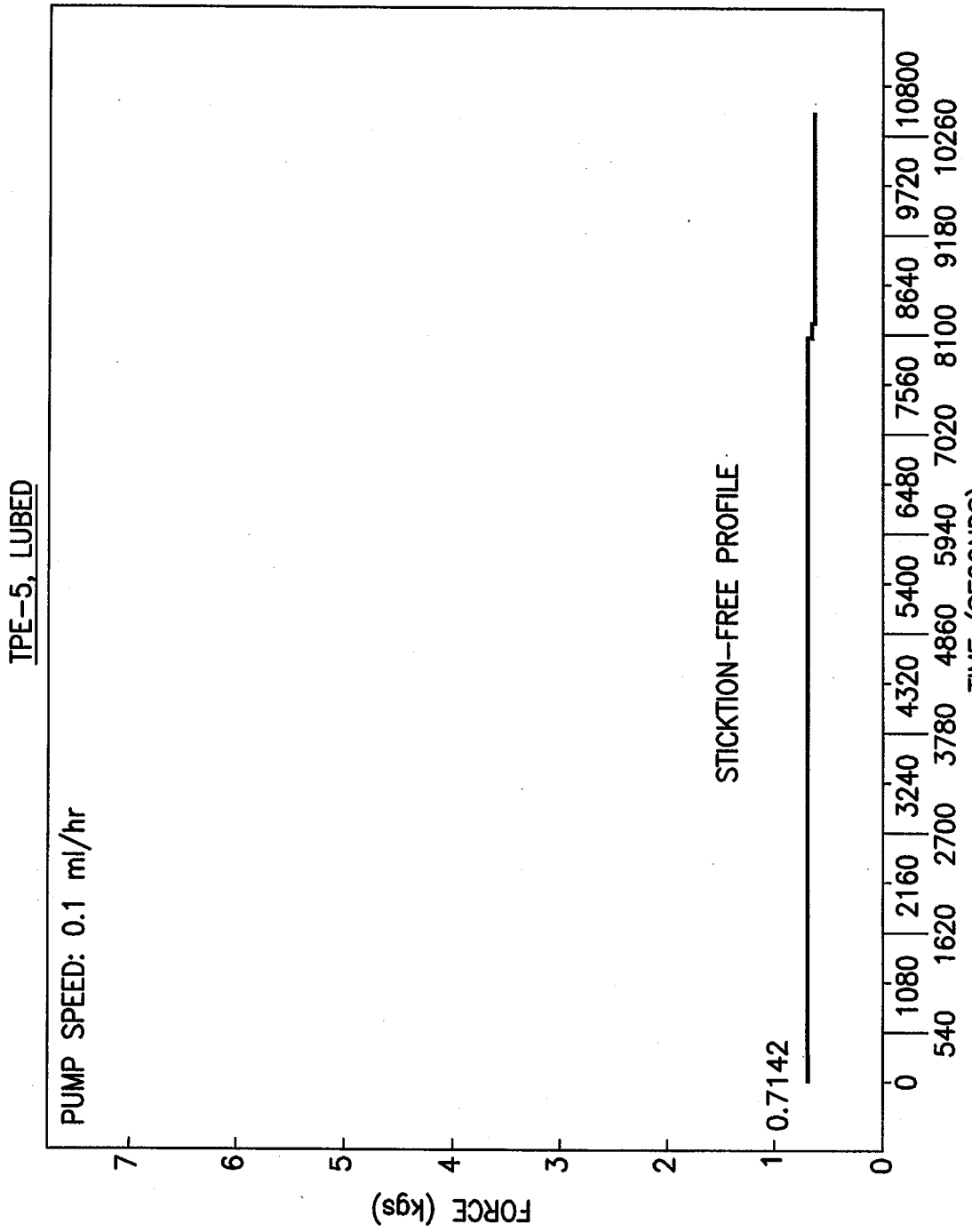
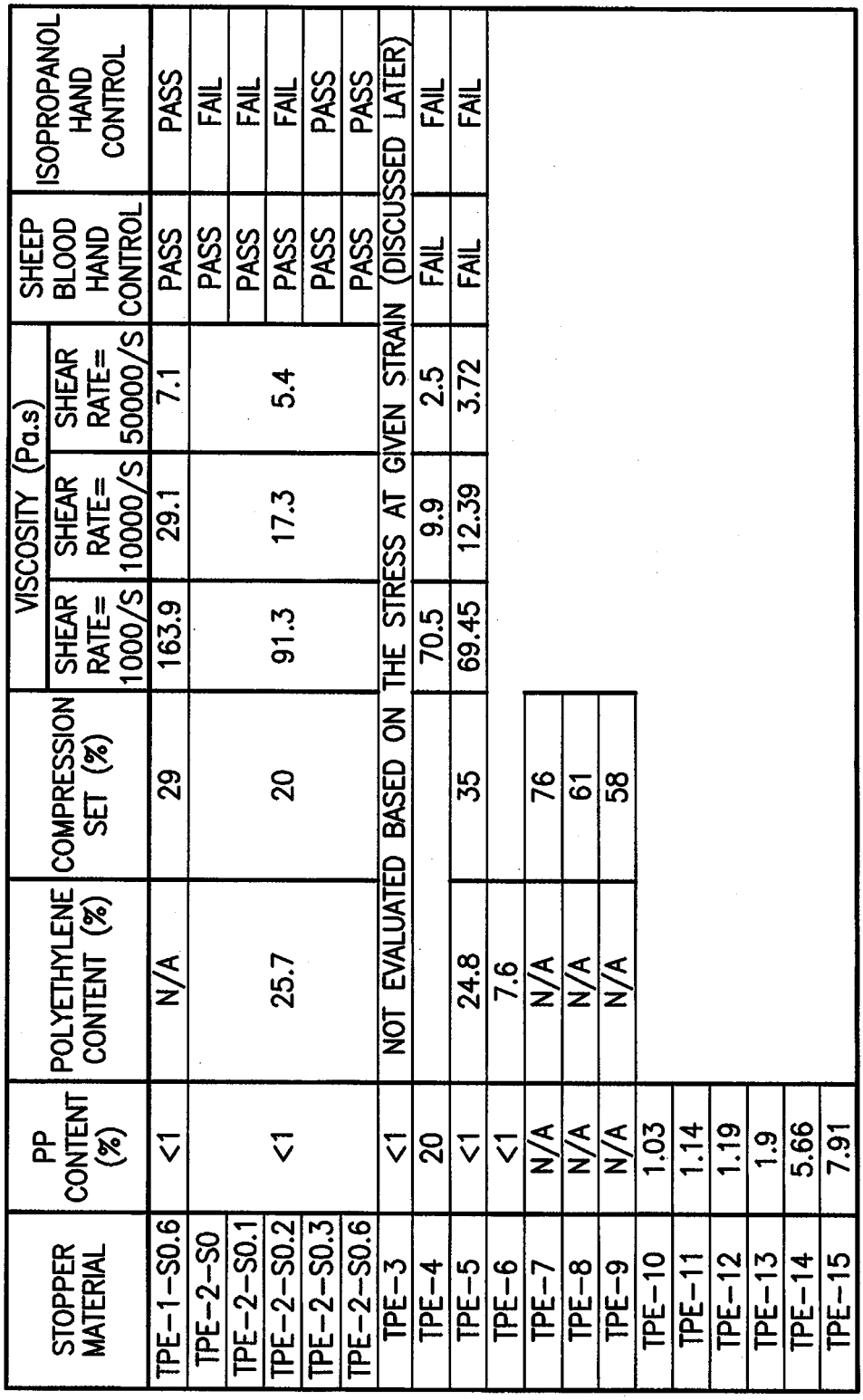

TPE-5, LUBED
PUMP SPEED: 0.1 ml/hr
STICKTION-FREE PROFILE
0.7142
FORCE (kgs)
0
1
2
3
4
5
6
7
0
540
1080
1620
2160
2700
3240
3780
4320
4860
5400
5940
6480
7020
7560
8100
8640
9180
9720
10260
10800
TIME (SECONDS)

FIG.19

STOPPER MATERIAL	PP CONTENT (%)	POLYETHYLENE CONTENT (%)	COMPRESSION SET (%)	LEAK PERFORMANCE (PASS/FAIL ISO7886-1 REQUIREMENT)
TPE-1-S0.6	<1	N/A	29	PASS
TPE-2-S0	<1	25.7	20	PASS
TPE-2-S0.1				PASS
TPE-2-S0.2				PASS
TPE-2-S0.3				PASS
TPE-2-S0.6				PASS
TPE-3	<1	NOT EVALUATED BASED ON THE STRESS AT GIVEN STRAIN (DISCUSSED LATER)		
TPE-4	20			
TPE-5	<1	24.8	35	PASS
TPE-6	<1	7.6		
TPE-7	N/A	N/A	76	FAIL
TPE-8	N/A	N/A	61	FAIL
TPE-9	N/A	N/A	58	FAIL
TPE-10	1.03			
TPE-11	1.14			
TPE-12	1.19			
TPE-13	1.9			
TPE-14	5.66			
TPE-15	7.91			

FIG.20



STOPPER MATERIAL	PP CONTENT (%)	POLYETHYLENE CONTENT (%)	COMPRESSION SET (%)	VISCOSITY (Pa.s)			SHEEP BLOOD HAND CONTROL	ISOPROPANOL HAND CONTROL
				SHEAR RATE= 1000/S	SHEAR RATE= 10000/S	SHEAR RATE= 50000/S		
TPE-1-S0.6	<1	N/A	29	163.9	29.1	7.1	PASS	PASS
TPE-2-S0	<1	25.7	20	91.3	17.3	5.4	PASS	FAIL
TPE-2-S0.1							PASS	FAIL
TPE-2-S0.2							PASS	FAIL
TPE-2-S0.3							PASS	PASS
TPE-2-S0.6							PASS	PASS
TPE-3	<1	NOT EVALUATED BASED ON THE STRESS AT GIVEN STRAIN (DISCUSSED LATER)						
TPE-4	20			70.5	9.9	2.5	FAIL	FAIL
TPE-5	<1	24.8	35	69.45	12.39	3.72	FAIL	FAIL
TPE-6	<1	7.6						
TPE-7	N/A	N/A	76					
TPE-8	N/A	N/A	61					
TPE-9	N/A	N/A	58					
TPE-10	1.03							
TPE-11	1.14							
TPE-12	1.19							
TPE-13	1.9							
TPE-14	5.66							
TPE-15	7.91							

FIG.21

TPE-2 WITH DIFFERENT ERUCAMIDE LEVEL, UNLUBED STOPPER	BREAK-LOOSE FORCE (lbf), AVG/ ST DEV, IT16-01 (SPEED: 1150 ml/hr)	BREAK-OUT FORCE (lbf), AVG/ ST DEV, IT16-01 (SPEED: 1150 ml/hr)	SUSTAINING FORCE (lbf), AVG/ ST DEV, IT16-01 (SPEED: 1150 ml/hr)	PUMP FORCE (lbf), AVG/ST DEV, IT1687-02 (SPEED: 10 ml/hr)
0%	3.07/0.17	0.8/0.19	0.45/0.11	2.17/0.31
0.1%	3.05/0.17	0.73/0.20	0.44/0.08	1.76/0.15
0.2%	2.92/0.14	0.59/0.35	0.35/0.11	1.51/0.24
0.3%	2.89/0.17	0.50/0.19	0.34/0.08	1.25/0.18

FIG.22

TPE	LEAK PRESSURE RANKING	SUSTAINING FORCE RANKING
TPE-1-S0.6	2	2
TPE-2-S0.6	1	3
TPE-3	3	1

FIG.23

TPE	LEAK PRESSURE (psi), AVG/ST DEV, IT1499-01	SUSTAINING FORCE (lbf), AVG/ST DEV, IT16-01
TPE-1-S0.6	227.4/35.4	1.03/0.09
TPE-2-S0.6	245.3/32.5	1.14/0.05
TPE-3	121.8/17.7	0.73/0.08

FIG.24

STOPPER MATERIAL	PP CONTENT (%)	POLYETHYLENE CONTENT (%)	COMPRESSION SET (%)	VISCOSITY (Pa.s)			SHEEP BLOOD HAND CONTROL	ISOPROPANOL HAND CONTROL	STRESS AT GIVEN STRAIN
				SHEAR RATE= 1000/S	SHEAR RATE= 10000/S	SHEAR RATE= 50000/S			
TPE-1-S0.6	<1	N/A	29	163.9	29.1	7.1	PASS	PASS	PASS
TPE-2-S0	<1	25.7	20	91.3	17.3	5.4	PASS	FAIL	
TPE-2-S0.1							PASS	FAIL	
TPE-2-S0.2							PASS	FAIL	
TPE-2-S0.3							PASS	PASS	PASS
TPE-2-S0.6							PASS	PASS	PASS
TPE-3	<1	NOT EVALUATED BASED ON THE STRESS AT GIVEN STRAIN							FAIL
TPE-4	20			70.5	9.9	2.5	FAIL	FAIL	
TPE-5	<1	24.8	35	69.45	12.39	3.72	FAIL	FAIL	
TPE-6	<1	7.6							
TPE-7	N/A	N/A	76						
TPE-8	N/A	N/A	61						
TPE-9	N/A	N/A	58						
TPE-10	1.03								
TPE-11	1.14								
TPE-12	1.19								
TPE-13	1.9								
TPE-14	5.66								
TPE-15	7.91								

FIG.25

TPE STOPPER IN DESIGN-5, 10ml SYRINGE, UNLUBED STOPPER, E-BEAM STERILE SYRINGE, NO NEEDLE ATTACHED, SLIP ADDITIVE (ERUCAMIDE) LEVEL IN TPE-2 (%)	ISOPROPANOL HAND CONTROL	SHEEP BLOOD HAND CONTROL
0%	BAD CONTROL WITH SPURTING	GOOD CONTROL AT DROPLET LEVEL
0.1%	BAD CONTROL WITH SPURTING	GOOD CONTROL AT DROPLET LEVEL
0.2%	BAD CONTROL WITH SPURTING	GOOD CONTROL AT DROPLET LEVEL
0.3%	GOOD CONTROL AT DROPLET LEVEL	GOOD CONTROL AT DROPLET LEVEL

FIG.26

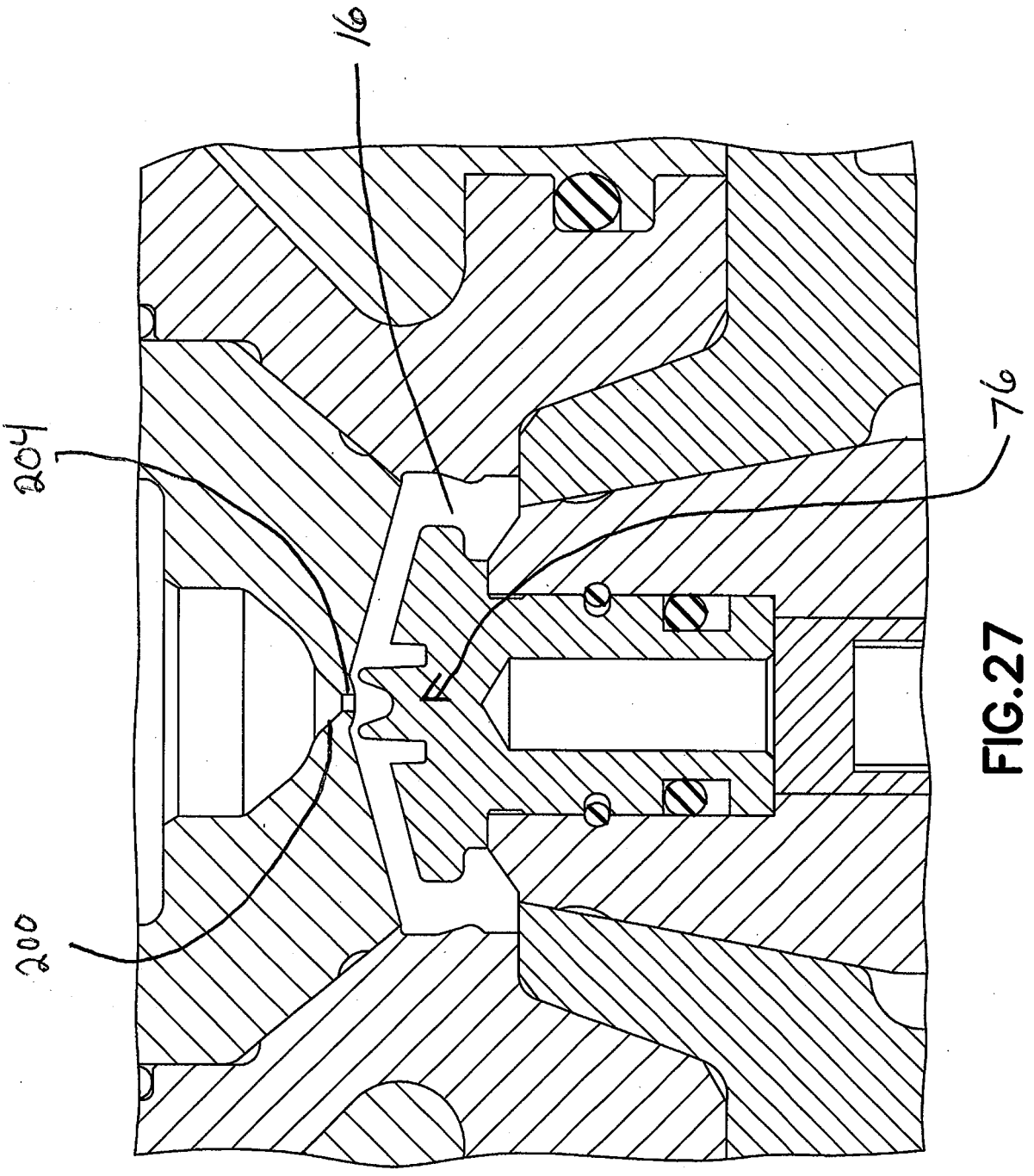


FIG.27

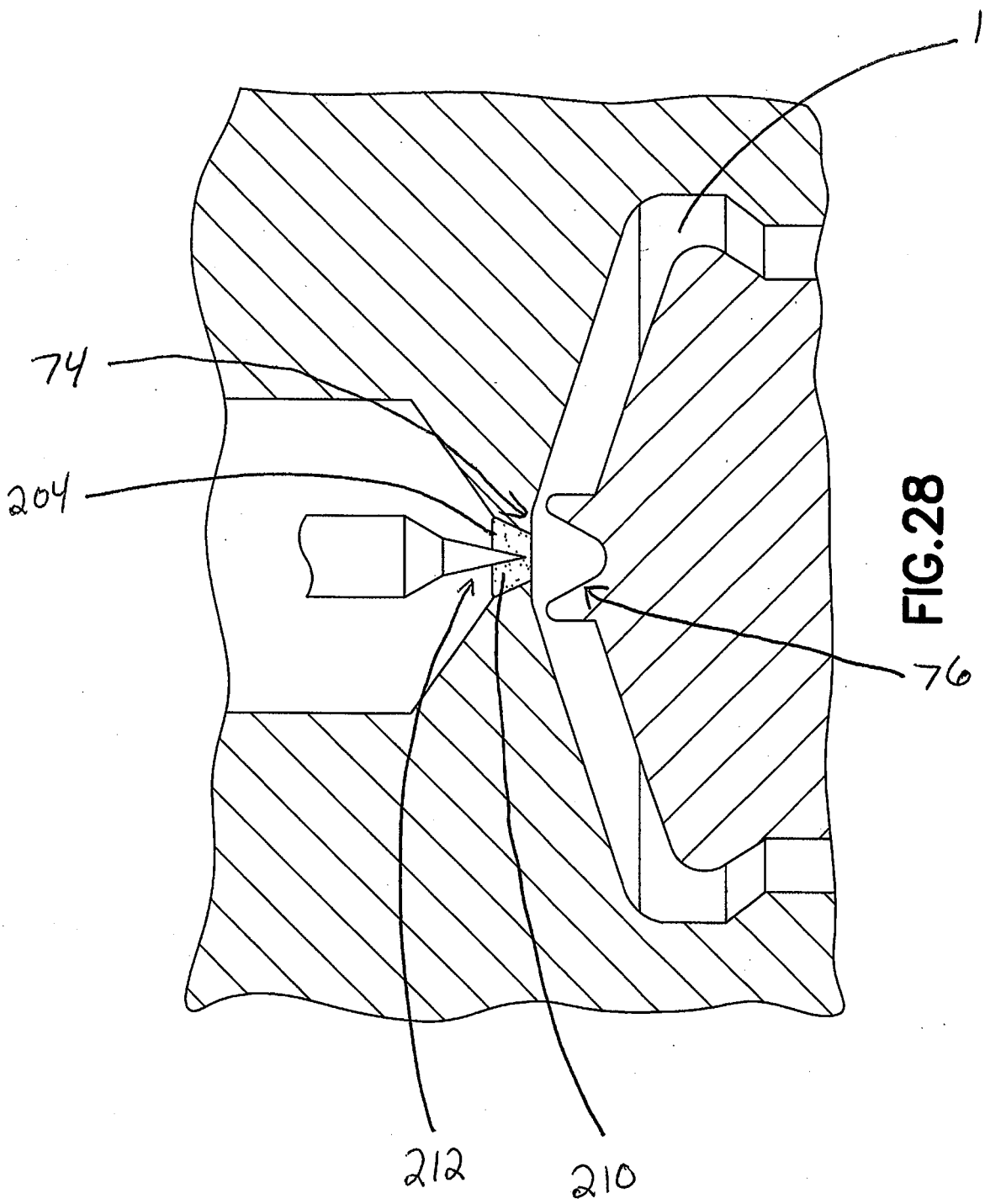


FIG.28