(12) UK Patent Application (19) GB (11) 2 351 086 (13) A

(43) Date of A Publication 20.12.2000

(21) Application No **9901749.3**

(22) Date of Filing **28.01.1999**

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(51) INT CL[7]
A61K 6/02

(52) UK CL (Edition R)
C3V VBC VET

(56) Documents Cited
Scand J Dent Res No. 92(2), 1984, BRUNE et al, "Initial Acid of Dental Cements", pages 156-160

(58) Field of Search
UK CL (Edition R) **C3V VAM VBC VET**
INT CL[7] **A61K 6/00 6/02 6/083**
CHABS, EPODOC, JAPIO, WPI.

(54) Abstract Title
Infrared radiation curing of ionomer cements

(57) Dental ionomer cements are cured using infrared radiation at wavelengths ranging 0. 1-100 μm. This results in dental ionomer cements having accelerated setting speeds, surface hardness and accelerated resistance to water contamination. Further the dental ionomer cements may be polished immediately following the infrared curing. Further disclosed is the use of infrared radiation consecutively or simultaneously with UV radiation for curing dental ionomer cements.

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CURING OF CEMENTS

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The device and concept of effecting (e.g. accelerating) the set of acid-base, especially "ionomer" cements, (union of metal oxides and glasses with polymers in the presence of water) to achieve up to a 10-fold improvement in their "instantaneous" surface hardness. Cement formulations curable by this means include all those that contain ionomer (acid-base) components. These are the so-called conventional, resin-modified, resin-based and all other resinous ionomer-containing formulations. The improvements derivable from the IR curing include the possibility of finishing/polishing ionomer cements and the increased resistance to water-solubility immediately following IR curing. These are particularly desirable in dental formulations of conventional, non-resin-based glass-ionomer cements.

NOVEL CURING OF IONOMER CEMENTS

Ionomer cements have found use in dentistry as adhesive restorative materials. Ionomer is the term coined for the reaction between basic glasses or oxides and ionic polymers. Commonly employed ionic polymers include poly(alkenoic acid)s, poly(vinyl phosphonic acid) and copolymers of these two acid types. These cements set automatically once the glass has been mixed with the suitable polymer in the presence of water. The cements formed from glass in this way were later termed Conventional glass-ionomer cements. These cements, introduced into the market in the mid 1970s, usually achieve a state of clinically set over a period of 4 - 6 minutes from the onset of mix. They also have working times of 2 - 4 minutes, during which time the cement can be manipulated, into the dental cavity. The later day version of the glass-ionomer cements were resin-modified formulations that attained clinical set on "command" from the energy of blue light (400 nm - 500nm). These cements had the advantages of: i) variable working time to suit dental practitioner and procedure; ii) low water susceptibility due to the presence of the resin and the fast attainment of clinical set.

This invention relates to the use of infrared radiation (IR) as a suitable source of energy for accelerating the set of ionomer cements. In this way, it is similar to the existing blue light. However, unlike the blue light, which causes electronic transitions in the outer valence shells of photoinitiators chemicals, the IR causes the vibrational motion of atoms, molecules and chain segments of the components of the ionomer cements. These include the constituents of the: i) atoms of the glass Al, Ca, Si, etc; and ii) parts of the acidic groups of the polymers and other acids, C=O, O-H, P=O, etc. In this way, Infrared radiation (0.1 micron - 100 micron) but preferable (3 microns - 15 microns) will energise the parts of the ionomer cements directly involved in the acid/base reaction. This translates to a noticeable increase in the speed of set of the cements. Since these cements progressively become less susceptible to water contamination as they set, the IR curing of ionomer cements also reduces the vulnerability of freshly mixed cements to water attack. The IR cured cements also take-up polish more readily due to their accelerated development of hardness. Therefore, IR is a means of replicating the advantages of the existing light curing of ionomer cements. It also has the added advantage of also being applicable to non-resin-based formulations.

The IR curing of ionomer cements can be demonstrated for all formulations containing the ionomer constituents. These include the reaction products of glasses, oxides and other precursors of the constituent metal atoms of ionomer cements - Ca, Si, O, F, Zn, Mg, Al, and other suitable tri- and divalent atoms in cement formulations. Examples of the other component of the formulations are: i) alkenoic acid(s); ii) phosphonic acid(s); iii) sulphonic acid(s); iv) any combinations in the form of physical blends and copolymers of the preceding acids. Also included are ionomer cements in resin-modified, resin incorporated and formulations containing polymerisable "half ester- half acid" chemicals and photoinitiators.

The invention claims right on the concept of using IR, particularly of the range (3-15 microns) to accelerate the set of acid-base cements. We also include the use of IR radiation in both resinous, resin included and resin-modified formulations as well as the Conventional acid-base cements. Finally, the use of IR, either as the only source of external activation of ionomers or consecutively or simultaneously with any other energy source, e.g. blue light is covered by our invention. The invention will now be described by way of examples of experimental data.

The state and speed of the acid-base reaction of ionomer cements can be followed by measuring the pH of the setting cement pastes. The pH of ionomer cements starts from about 1.5 and then rises rapidly at first and then more slowly, as cements set. The viscosity and the hardness of the cements also follow this trend. At the same time, the resistance to water dissolution increases. Therefore, pH is a measure of the efficacy of the IR curing, while improved surface hardness, early take-up of polish and resistance to water dissolution are the measurable deliverables and criteria for confirming the usefulness of the IR curing of ionomer cements.

A number of components necessary to build an infrared pulsing device were purchased from L.O.T Oriel Instruments, Stratford, Connecticut, USA. The two important components of the device were the illuminator and the IR element. The illuminator consisted of:

1. A built-in power supply with the current regulated output specifically designed for the IR element
2. A soft-start regime when the power is applied to avoid "socking" the IR element
3. A precision 10-turn output current control and digital display of the current and voltage

The IR element used was a high temperature, oxidation resistance wire. The coil was supported on a grooved alumina support mandrel. The radiating area was 0.14 inches (3.6 mm) wide and 0.14 inches (3.6 mm) long.

pH Measurements

With a current setting of 1.80 amps and 4.5 V, the effect of distance of IR element from the surface of the cements being cured was investigated using pH measurements. In these experiments cement comprising powder [100 parts glass, 10 parts poly(acrylic acid) and 3 parts tartaric acid] and liquid [25 parts poly(acrylic acid), 12.5 parts tartaric acid and 62.5 parts water] were mixed at 4:1 ratio. The cements (termed KG23/5) were mixed and the experiment conducted at room temperature (19°C). Cements were mixed and placed in a thin layer at the tip of a Flat head Combination BDH pH electrode connected to an Ionmeter. In this experiments, the cements at the end of the electrode were placed at requisite distance from the IR element and irradiation commenced at t = 90s from the onset of mix. The results of these experiments are presented in Table 1. From the Table, the efficacy of the IR curing can be observed. IR curing (for all the cements from 1 cm to 8 cm distance from the IR element) increased the rate of rise in the pH of the cements. Not surprisingly, the closer the cement was to the IR element, the more efficient the IR curing was.

Following these experiments, the effect of IR curing on the pH of various commercial dental ionomer cements was determined. These cements included Fuji IX and Fuji II LC [Ex. GC Corp, Japan] and Diamond Carve [Ex. Kemdent, UK]. Fuji IX is a polycarboxylate-based Conventional cement. Diamond Carve is a polyphosphonate-based Conventional cement, while Fuji II LC is a resin-modified glass-ionomer cement, with carboxylic acid ionomer content. The pHs of these cements, IR cured and otherwise, were determined using a regime of 1.8 amps (4.5V). Cements were cured from a distance of 4 cm from the cement surface and from t = 90s from the onset of mix. The Fuji II LC was light cured for 40s. Table 2 presents the results of these experiments. In Table 2, "F IX" is Fuji IX, "F LC" is Fuji II LC and "DC" is Diamond Carve. As can be observed from Table 2, IR curing was effective in increasing the speed of pH increase in all the setting pastes of the cements evaluated. These results suggest that IR curing is effective in all the classes of the commercial ionomer cements (glass polyalkenoates and glass polyphosphonates) and in resin-modified ionomer cements.

Solubility Measurements

The solubility of the experimental cement (KG23/5) was determined as a function of distance from the IR element. These experiments were carried out at ambient temperature (19°C) and using an IR curing regime of 1.8A (4.5V) and curing time of 60s (90-150s from the onset of mix). The solubility measurements were made with the aid of a JENWAY Total Dissolved Solids (TDS) meter (model 4076). 1 gram of cement was mixed and placed in stainless steel rings of 13-mm diameter by 2-mm height. These cement discs were then subjected to IR curing and placed at the bottom of glass vials containing 10.00 g of distilled water at time t = 3.00 ± 0.20 minutes from the onset of mix. The TDS of the distilled water had been pre-determined as 4.3 mg/l. This value has been subtracted from the readings of the cement containing aqueous solutions in the readings presented in Table 3. The TDS of the solutions increase as solids dissolve from the cement into the water. Therefore, the improvement in the resistance to water dissolution of the ionomer experimental cement is obvious from the low TDS values of cements upon IR curing. As indicated from the pH measurements of Table 1, the efficiency of the IR irradiation decreases with distance away from the IR element.

The solubilities of commercial dental cements were also determined in the manner described. The effect of IR curing on the solubilities of Fuji IX, Fuji II LC, KG23/5 and Diamond Carve is presented in Table 4. The solubilities of all the cements evaluated reduced with IR irradiation. Noteworthy is the fact that the solubility of Fuji II LC was significantly improved by the IR curing. In the experiments, Fuji II LC was light cured on one side and its solubility evaluated. In the IR experiment, Fuji II LC was first light cured for 40s and then IR cured for 60s. The reduction in the susceptibility to water of Fuji II LC at the point of introduction (t= 3.00 minutes) is due to the fact that the acid-base reaction of the cement has been deliberately retarded to accommodate the light curing reaction. From the results of the experiment, it appears desirable to IR cure light cure, resin-modified ionomer cements after the initial light curing.

Mechanism of IR curing - Temperature Effect

In an attempt to judge the efficacy and to explore the mechanism of the IR curing of the ionomer cements, a series of experiments were undertaken. These included: i) evaluating the temperature increase of 1.00g of cement upon light curing; and ii) evaluating the hardness of cements isothermally, achieved via IR curing and also by placing cements in pre-heated ovens.

The temperature rise of 1.00g of cements measured at the IR cured surface with the aid of a k-type thermocouple connected to a Digitron Instrument ltd thermometer were determined as function of distance of the cements from the IR element. IR curing commenced at t = 90s. The IR curing regime employed was 1.80 amps (4.5 V). The results of these experiments are contained in Table 5. From this Table, the increase in temperature with IR curing time and distance towards the IR element is clear. Although the temperature rose towards 100°C, after 60s of IR curing, i.e. at t = 150s, the temperature attained was less than 60 °C in all cases. The maximum temperature indicated for the curing of dental ionomer cements will be 40 - 60 s. This is in line with the current practice of 40 s blue light curing of resin-modified glass-ionomer cements. In order to judge the effect that this temperature rise had on the efficacy of the IR curing, Diamond Carve cements were mixed and flattened between two glass slides in the stainless steel rings. Cement samples were stored at 60°C to simulate the effect of IR irradiating the cement for 60 s from a distance of 1 cm from the cement surface. Cement samples were also subjected to this IR regime and stored at 37°C. Finally, cements were mixed and stored without IR curing at 37°C. All cements were exposed to 100 % rh environment at t = 5 minutes. The scratch hardness measured by a Leitz Vickers indenter was determined. From these measurements, made at t = 15, 30, 60, 180 and 300 minutes, the hardness of the cements were evaluated. The results of these experiments are presented in Table 6 and Figure 1.

The 15 and 30-minute hardness values of the IR cured cements are significantly superior to those of the non-IR-cured variants. This proves the efficacy of IR curing of ionomer cements. The fact that the hardness of all the cements, IR cured and otherwise, were eventually similar (after 3 hours) indicates that the IR curing is basically a "rate of reaction acceleration" phenomenon. Indeed, it is a surface phenomenon that has negligible effect on the bulk mechanical properties of ionomer cements. This is confirmed by the results presented in Table 7, giving details of the surface hardness and compressive strength of the experimental KG23/5 cement.

Surface Hardness Measurements

The efficacy of the IR curing of ionomer cements as a means of creating "command" cured dental cements can be clearly observed from Table 8 and Figure 2. The profiles for the surface hardness of Conventional and the current light cured resin-modified cements are typified by those of KG23/5 and Fuji II LC respectively in Table 8. While conventional glass-ionomer cements develop hardness sluggishly in a logarithmic fashion, light cure cements exhibit a linear trend, with a much higher "instantaneous" hardness than the Conventional cement variants. Also, in addition, the 15 minute value of the light cured cements is ~ 50 % the 24 hour (or final) hardness value. This is in contrast to the case with the Conventional cements with a scratch hardness value of 15 minutes/24 hours (0.01 value) of ~ 10 %. This is a main reason why conventional glass-ionomer cements do not take-up polish until they are mature, well over 24 hours old. The efficacy of the IR curing can therefore be measured by the attainment of the 50% "0.01" scratch hardness value. (Fuji II LC has a value of 53%).

Clearly, the IR regime employed on the KG23/5 cements (1.8 Amps and 60s from 1 cm from the cement surface) met this objective, with a value of 82 %. The effect of IR regime on the scratch hardness and the "0.01" value for the KG23/5 cement is presented in Table 9, 10 and Figure 3. From Table 10, the IR regimes that create command cure cements comparable to those currently existing are:

- 1 cm distance from IR element and 20-60 s IR curing
- 2 cm from the IR element and 40-60 s IR curing
- 3 cm from the IR element and 6-0 s IR curing

Finally, in a series of experiments, the efficacy of the IR curing of ionomer cements and the estimation of the IR affected area of cements were determined. This was achieved by placing the IR element underneath holes drilled at judicious distances apart into a 3-mm thick aluminium plate. Directly on top of these holes in a series of experiments, were placed cement discs resting on wooden rods. These cements were then irradiated with IR for 60s and with the IR element a distance of 1 cm or 2 cm away from the cement surface. In this way, the 3D map of the surface hardness of the cements placed over 1, 2, 4 and 6-mm diameter holes were determined. The 3D surface hardness map of cements IR cured for 60s, 1 cm away from the IR element and over the 4-cm hole is presented in Figure 4. The "mountain" topography was obtained by the careful mapping of the co-ordinates from which the scratch hardness values were obtained. In Figure 4, the scratch on the cement was created between 5 and 10 minutes from the onset of mixing the cement. The value on the x-axis is distance in mm. This shows that an area the shape of an ellipse of dimensions 7 mm by 5 mm had attained the maximum hardness of 450 MPa - 500 MPa.

TABLE 1. THE EFFECT OF DISTANCE FROM THE IR ELEMENT ON THE pHs OF KG23/5 CEMENTS

Cement age (s)	No IR	1 cm IR	2 cm IR	3 cm IR	4 cm IR	5 cm IR	6 cm IR	7 cm IR	8 cm IR
100	1.32	1.05	1.11	1.24	1.08	1.04	0.93	1.21	1.19
110	1.36	1.07	1.14	1.26	1.09	1.02	0.94	1.23	1.19
120	1.38	1.12	1.21	1.3	1.11	1.06	0.97	1.25	1.21
130	1.41	1.2	1.29	1.35	1.15	1.1	1	1.27	1.24
140	1.43	1.3	1.38	1.41	1.19	1.14	1.04	1.29	1.27
150	1.45	1.43	1.47	1.48	1.25	1.19	1.09	1.31	1.3
160	1.47	1.49	1.54	1.55	1.32	1.25	1.13	1.35	1.34
170	1.5	1.56	1.6	1.6	1.39	1.31	1.19	1.38	1.39
180	1.52	1.63	1.65	1.65	1.45	1.36	1.23	1.44	1.43
190	1.54	1.69	1.7	1.71	1.5	1.41	1.29	1.47	1.47
200	1.56	1.75	1.75	1.75	1.55	1.46	1.34	1.52	1.52
210	1.58	1.81	1.79	1.79	1.6	1.51	1.38	1.56	1.57
220	1.6	1.86	1.84	1.84	1.65	1.54	1.42	1.59	1.6
230	1.62	1.92	1.88	1.87	1.69	1.58	1.46	1.64	1.63
240	1.64	1.97	1.92	1.9	1.73	1.61	1.5	1.67	1.66
250	1.66	2.03	1.96	1.93	1.77	1.66	1.53	1.7	1.7
260	1.67	2.08	1.99	1.96	1.82	1.69	1.55	1.72	1.72
270	1.69	2.13	2.03	1.99	1.84	1.72	1.59	1.75	1.75
280	1.71	2.17	2.07	2.02	1.87	1.75	1.62	1.78	1.78
290	1.73	2.22	2.1	2.05	1.9	1.78	1.64	1.8	1.8
300	1.74	2.26	2.14	2.08	1.94	1.81	1.67	1.83	1.82
310	1.76	2.31	2.18	2.1	1.97	1.84	1.7	1.85	1.84
320	1.77	2.35	2.22	2.14	2	1.87	1.72	1.87	1.87
330	1.79	2.39	2.25	2.16	2.03	1.9	1.75	1.89	1.89
340	1.8	2.42	2.29	2.19	2.06	1.92	1.78	1.91	1.91
350	1.82	2.45	2.32	2.22	2.09	1.95	1.8	1.93	1.92
360	1.83	2.48	2.35	2.24	2.12	1.98	1.83	1.96	1.95
370	1.84	2.51	2.38	2.27	2.15	2	1.85	1.97	1.97
380	1.86	2.54	2.41	2.29	2.17	2.02	1.87	1.99	1.98
390	1.87	2.57	2.43	2.32	2.2	2.05	1.9	2.01	2
400	1.89	2.6	2.46	2.34	2.23	2.07	1.92	2.03	2.02
410	1.9	2.63	2.49	2.37	2.26	2.1	1.95	2.05	2.04
420	1.9	2.65	2.51	2.4	2.28	2.12	1.97	2.07	2.06
430	1.92	2.68	2.54	2.42	2.3	2.15	1.99	2.09	2.07
440	1.92	2.71	2.56	2.44	2.33	2.17	2.01	2.11	2.09
450	1.93	2.73	2.58	2.46	2.35	2.21	2.04	2.13	2.11
460	1.95	2.76	2.61	2.48	2.38	2.23	2.07	2.15	2.13
470	1.95	2.79	2.63	2.51	2.4	2.25	2.09	2.16	2.15
480	1.96	2.84	2.65	2.53	2.42	2.27	2.12	2.18	2.16
490	1.97	2.86	2.67	2.55	2.44	2.29	2.14	2.2	2.18
500	1.98	2.88	2.68	2.57	2.46	2.31	2.17	2.21	2.19
510	1.99	2.9	2.7	2.58	2.48	2.34	2.21	2.23	2.2
520	1.99	2.92	2.71	2.6	2.5	2.35	2.24	2.25	2.23
530	2	2.93	2.73	2.63	2.52	2.38	2.26	2.26	2.25
540	2.01	2.94	2.74	2.64	2.54	2.39	2.29	2.28	2.26
550	2.02	2.96	2.76	2.66	2.56	2.4	2.32	2.3	2.27
560	2.02	2.97	2.78	2.68	2.58	2.43	2.35	2.31	2.29
570	2.03	2.98	2.8	2.69	2.59	2.45	2.37	2.33	2.31
580	2.04	2.99	2.82	2.71	2.61	2.47	2.4	2.35	2.31
590	2.04	3	2.83	2.73	2.63	2.49	2.42	2.36	2.33
600	2.05	3.01	2.85	2.75	2.64	2.5	2.44	2.37	2.34

TABLE 2. THE EFFECT OF IR CURING ON THE pHs OF GLASS-IONOMER CEMENTS

Cement age (s)	F IX	F IX - IR	F LC	F LC - IR	F LC - LC/IR	DC	DC - IR
100	1.4	1.18	1.91	1.96	1.78	1.7	1.6
110	1.42	1.18	1.93	1.95	1.82	1.73	1.62
120	1.44	1.23	1.95	1.98	1.89	1.75	1.67
130	1.47	1.28	1.96	2.03	1.95	1.78	1.74
140	1.49	1.35	1.98	2.11	2.01	1.81	1.81
150	1.52	1.44	2	2.2	2.05	1.84	1.9
160	1.55	1.53	2.03	2.29	2.09	1.86	1.98
170	1.59	1.63	2.05	2.34	2.12	1.89	2.02
180	1.62	1.71	2.08	2.4	2.15	1.92	2.07
190	1.65	1.8	2.1	2.43	2.16	1.94	2.12
200	1.68	1.89	2.13	2.47	2.18	1.97	2.17
210	1.71	1.96	2.16	2.52	2.2	2	2.2
220	1.74	2.03	2.19	2.56	2.22	2.02	2.25
230	1.78	2.1	2.22	2.6	2.23	2.04	2.3
240	1.81	2.17	2.25	2.63	2.25	2.06	2.34
250	1.84	2.24	2.28	2.65	2.26	2.08	2.38
260	1.87	2.3	2.31	2.67	2.27	2.1	2.42
270	1.9	2.37	2.33	2.68	2.28	2.13	2.45
280	1.92	2.42	2.36	2.7	2.29	2.14	2.49
290	1.95	2.48	2.38	2.73	2.31	2.15	2.52
300	1.98	2.54	2.4	2.74	2.32	2.17	2.55
310	2.01	2.6	2.42	2.75	2.32	2.18	2.59
320	2.03	2.67	2.44	2.75	2.33	2.2	2.62
330	2.06	2.77	2.46	2.77	2.34	2.21	2.65
340	2.08	2.85	2.47	2.79	2.36	2.22	2.67
350	2.1	2.91	2.49	2.8	2.37	2.23	2.7
360	2.13	2.97	2.5	2.81	2.37	2.24	2.73
370	2.15	3.01	2.51	2.82	2.38	2.26	2.75
380	2.17	3.07	2.53	2.83	2.39	2.27	2.77
390	2.2	3.12	2.54	2.84	2.4	2.28	2.79
400	2.22	3.17	2.55	2.85	2.42	2.29	2.82
410	2.24	3.21	2.56	2.86	2.41	2.3	2.83
420	2.26	3.24	2.57	2.87	2.43	2.31	2.86
430	2.28	3.27	2.58	2.88	2.44	2.31	2.88
440	2.3	3.31	2.59	2.89	2.46	2.32	2.9
450	2.32	3.33	2.6	2.9	2.48	2.33	2.92
460	2.34	3.36	2.61	2.91	2.49	2.34	2.94
470	2.36	3.38	2.62	2.91	2.52	2.35	2.95
480	2.38	3.4	2.62	2.92	2.54	2.36	2.96
490	2.4	3.42	2.63	2.94	2.58	2.37	2.96
500	2.42	3.44	2.64	2.95	2.59	2.38	2.97
510	2.44	3.46	2.64	2.98	2.59	2.38	3
520	2.46	3.48	2.65	3.01	2.61	2.39	3.01
530	2.48	3.49	2.66	3.03	2.61	2.4	3.03
540	2.5	3.5	2.66	3.05	2.65	2.41	3.03
550	2.51	3.51	2.67	3.09	2.67	2.42	3.05
560	2.53	3.52	2.68	3.12	2.68	2.42	3.05
570	2.55	3.54	2.68	3.14	2.71	2.43	3.06
580	2.57	3.55	2.69	3.15	2.73	2.44	3.09
590	2.58	3.56	2.69	3.16	2.74	2.45	3.1
600	2.6	3.57	2.7	3.17	2.74	2.46	3.1

TABLE 3. THE EFFECT OF DISTANCE ON THE SOLUBILITY OF KG23/5 CEMENTS

Cement age (s)	1.0 cm	2.0 cm	3.0 cm	4.0 cm	5.0 cm	No IR
180						
200		3.6	12.3	13.4	15.4	70.4
220	2.2	3.8	12.4	14.3	15.8	70.8
240	2.4	4	12.5	15.1	16	69.8
260	2.5	4.1	12.9	15.6	16.1	69.4
280	2.6	4.3	13.4	15.9	16.3	69.5
300	2.7	4.5	13.8	16.1	16.5	95.1
320	2.7	4.6	14.1	16.3	16.8	117
340	2.9	4.7	14.3	16.4	16.9	128.5
360	3.1	4.7	14.4	16.5	17	142.8
380	3.3	4.8	14.4	16.6	17.1	150.1
400	3.4	4.9	14.4	16.6	17.3	151.3
420	3.5	4.9	14.4	16.5	17.3	153.8
440	3.6	4.9	14.4	16.5	17.4	155.8
460	3.6	5	14.4	16.4	17.5	157.4
480	3.6	5	14.4	16.3	17.6	158
500	3.6	5	14.5	16.2	17.7	159.8
520	3.6	5.1	14.5	16	17.8	161
540	3.6	5.1	14.5	16	17.9	162
560	3.5	5.1	14.6	15.9	18	165.7
580	3.5	5.2	14.6	15.8	18.1	169
600	3.5	5.2	14.6	15.7	18.2	171
660	3.4	5.2	14.6	15.6	18.5	174
720	3.4	5.2	14.7	15.6	18.7	177.5
780	3.4	5.3	14.7	15.5	19	180
840	3.4	5.3	14.7	15.4	19.1	182.5
900	3.4	5.3	14.6	15.4	19.3	184.6
960	3.4	5.3	14.6	15.3	19.5	186.7
1020	3.4	5.3	14.8	15.2	19.5	190.7
1080	3.4	5.3	14.8	15.2	19.6	194
1140	3.4	5.3	14.8	15.1	19.7	197.5
1200	3.4	5.3	14.8	15.1	20.1	212
1260	3.4	5.4	14.8	15	20.1	224
1320	3.4	5.4	14.8	15	20.1	227
1380	3.4	5.4	14.8	14.9	20.2	230
1440	3.4	5.4	14.5	14.9	20.2	232
1500	3.4	5.4	14.7	15	20.3	235
1560	3.4	5.4	14.8	15	20.3	237
1620	3.5	5.4	14.9	14.9	20.3	239
1680	3.5	5.4	14.6	14.8	20.3	241
1740	3.5	5.4	14.5	14.8	20.4	242
1800	3.5	5.4	14.5	14.8	20.4	244

TABLE 4. THE EFFECT OF IR CURING ON THE SOLUBILITY OF GLASS-IONOMER CEMENTS

Cement Age (s)	SOLUBILITY (mg/l)							
	Fuji II LC (40s LC+IR)	Fuji IX-IR	DC-IR	KG23/5-IR	Fuji II LC (40s LC)	Fuji IX	KG23/5	DC
240	9.2	5.5	6.4	11.7	62	56	69.8	24.4
260	9.5	5.6	6.9	11.2	63.7	61	69.4	24.7
280	9.6	6	7.31	11.7	65	65	69.5	24.9
300	9.7	8	8	12.7	66	67.5	95.1	25.1
320	9.9	8.1	8.5	13.4	66.1	69.5	117	25.3
340	9.9	8.3	8.8	14.3	66.1	70.9	128.5	25.4
360	10.1	8.5	8.9	15.1	65.8	71.9	142.8	26
380	10.2	8.6	9	15.6	65.5	72.8	150.1	26.2
400	10.3	8.8	9	15.9	65	73.5	151.3	26
420	10.3	8.9	9.1	16.1	64.6	74.2	153.8	26.1
440	10.3	9.1	9.1	16.3	64.2	74.7	155.8	26.1
460	10.3	9.3	9.1	16.4	63.8	75.2	157.4	26.1
480	10.3	9.5	9.2	16.5	63.5	75.6	158	26.4
500	10.3	9.6	9.2	16.6	63.1	76.1	159.8	26.5
520	10.3	9.8	9.2	16.6	62.8	76.4	161	26.6
540	10.2	9.8	9.2	16.5	62.5	76.8	162	26.8
560	10.2	9.9	9.2	16.5	62.2	77.2	165.7	27
580	10.2	10	9.2	16.4	61.9	77.5	169	27.3
600	10.2	10.1	9.2	16.3	61.6	77.8	171	27.5
660	10.3	10.4	9.1	16.2	60.8	78.9	174	28.3
720	10.3	10.6	9.1	16	60.1	80.1	177.5	28.8
780	10.3	10.7	9	16	59.6	81.2	180	29.3
840	10.3	10.9	9	15.9	59.2	82	182.5	29.5
900	10.3	11	8.9	15.8	58.8	82.7	184.6	29.8
960	10.3	11	8.9	15.7	58.6	83.4	186.7	30
1020	10.3	11.3	8.8	15.6	58.2	84	190.7	30.2
1080	10.3	11.4	8.8	15.6	57.9	84.6	194	30.4
1140	10.3	11.5	8.8	15.5	57.7	85.3	197.5	30.6
1200	10.3	11.6	8.8	15.4	57.5	85.8	212	30.8
1260	10.3	11.7	8.8	15.4	57.3	86.3	224	30.9
1320	10.3	11.8	8.8	15.3	57.1	86.8	227	31.1
1380	10.3	11.9	8.8	15.2	57	87.7	230	31.3
1440	10.3	12	8.8	15.2	56.9	87.7	232	31.5
1500	10.3	12	8.8	15.1	56.8	88.3	235	31.6
1560	10.3	12.1	8.8	15.1	56.7	88.7	237	31.8
1620	10.3	12.1	8.8	15	56.7	89.1	239	31.9
1680	10.3	12.2	8.8	15	56.5	89.5	241	32.1
1740	10.3	12.2	8.7	14.9	56.5	89.9	242	32.2
1800	10.3	12.3	8.7	14.9	56.5	90.3	244	32.3

Cements introduced into 10g of water at t=170s, only one surface of the disc is exposed to water.
The results have the 12.7 mg/l tds of water

TABLE 5. THE TEMPERATURE OF KG23/5 CEMENTS DURING IR PULSING
(Cement, pulsed with IR at 1.8 Amps, starting from t= 90s, with the probe at the surface of the cement)

Cement age (s)	1cm	2cm	3cm	4cm	5cm	6cm	7cm	8cm
120	***32***	***38.5***	***36.8***	***38.2***	***36.4***	***39.7***	***35.6***	***34.9***
130	***39.6***	***44***	***42.2***	***43.3***	***41.4***	***44.5***	***38.5***	***38.7***
140	***48.6***	***50***	***47.7***	***48.4***	***47***	***49.6***	***42.3***	***41.9***
150	***57.7***	***56.1***	***53.2***	***54***	***52.6***	***54.9***	***45.5***	***44.9***
160	66.7	62.5	59	59.7	58.7	60.5	48.5	47.9
170	79.7	69.2	64.7	65.1	64.1	66.3	52.1	50.9
180	88.2	74.7	70.1	70.9	69.8	71.5	54.9	53.3
190	94.9	78.9	74.8	76	74.7	76.5	57.9	56.2
200	103.8	81.7	78.2	80.1	78.4	80.1	60.9	58.9
210	108.1	83.8	80.9	82.9	81.7	82.7	63	61.5
220	110.2	85.1	82.9	85.9	84.1	84.4	64.9	63.6
230	111.6	86	84.2	87	85.7	85.7	66.7	65.7
240	112.7	86.5	85.2	88.3	86.9	86.9	68	67.2
250	113.4	86.5	85.7	89.6	87.9	87.6	69.2	68.5
260	113.8	86.6	86.2	91	88.6	88.1	70.2	69.3
270	113.7	86.8	86.6	92	88.9	88.7	70.6	70.1
280	112.4	86.7	86.7	92.1	89.1	89	70.7	70.6
290	109.4	86.7	86.8	92.3	89.4	89.4	70.7	71
300	108	86.7	87	92.3	89.3	89.6	71	71
310	107.2	86.3	87.1	91.9	89.5	89.7	71.2	71.1
320	106.5	86.2	87.2	92.3	89.4	89.9	71.4	71.1
330	105.9	85.8	87.2	92	89.5	90	71.4	70.9
340	105.4	85.7	87.3	91.4	89.3	89.7	71.3	70.8
350	104.4	85.6	87.5	90.9	89	89.6	71.3	70.7
360	104.4	85.3	87.6	90.6	89	89.6	71.1	70.4
370	103.6	85.3	87.6	90.5	88.9	89.5	70.9	70.1
380	103.4	85.2	87.6	89.6	88.7	89.5	70.8	70
390	103.2	85.2	87.8	89.2	88.5	89.6	70.5	69.6
400	103	85.1	88	89.1	88.6	89.7	70.3	69.6
410	102.9	85	88	89	88.5	89.7	70.2	69.1
420	102.8	84.6	87.7	88.5	88.4	89.8	69.9	69
430	102.7	84.4	87.7	88	88.2	89.7	69.8	68.6
440	102.8	84.4	87.9	87.8	88.3	89.8	69.8	68.3
450	102.8	84.4	87.8	87.7	88.3	89.9	69.8	68.2
460	102.8	84.2	87.7	88	88.2	90	69.8	67.8
470	102.9	84.2	87.6	87.8	88.4	89.9	69.7	67.8
480	102.9	84	87.5	87.8	88.3	89.9	69.5	67.7
490	102.9	83.7	87.6	87.7	88.3	89.6	69.4	67.5
500	102.9	83.8	87.7	87.6	88.1	89.2	69.1	67.4
510	103	84	87.7	87.7	88.1	89.1	68.9	66.9
520	103.1	84	87.6	87.5	88.2	89.1	69	66.4
530	103.2	84	87.6	87.2	88.3	89	68.9	66.3
540	103.3	84	87.6	87.1	88.3	88.8	68.8	66.3
550	103.4	84	87.6	87	88.6	88.7	69	65.9
560	103.5	83.9	87.5	87.3	88.5	88.7	68.9	65.9
570	103.7	83.9	87.5	87.5	88.4	88.5	69	65.6
580	103.8	83.8	87.4	87.4	88.3	88.4	69.1	65.6
590	104	84	87.3	87.5	88.2	88.7	69.1	65.2
600	104.2	84	87.3	87.5	88.2	88.7	69	65.1

TABLE 6. THE EFFICACY OF IR CURING OF KG23/5 CEMENT

Cement Age (mins)	DC-37 (Wet)	DC-60 (Wet)	DC-IR (37 Wet)
10	695.8	773.1	1229.1
30	669.6	957.7	1130.5
60	893.5	1165.7	1207.1
180	1181.3	1379.8	1090.4
300	1019	1234.5	1150.7

FIGURE 1. THE EFFECT OF IR CURING ON THE HARDNESS OF DIAMOND CARVE CEMENT

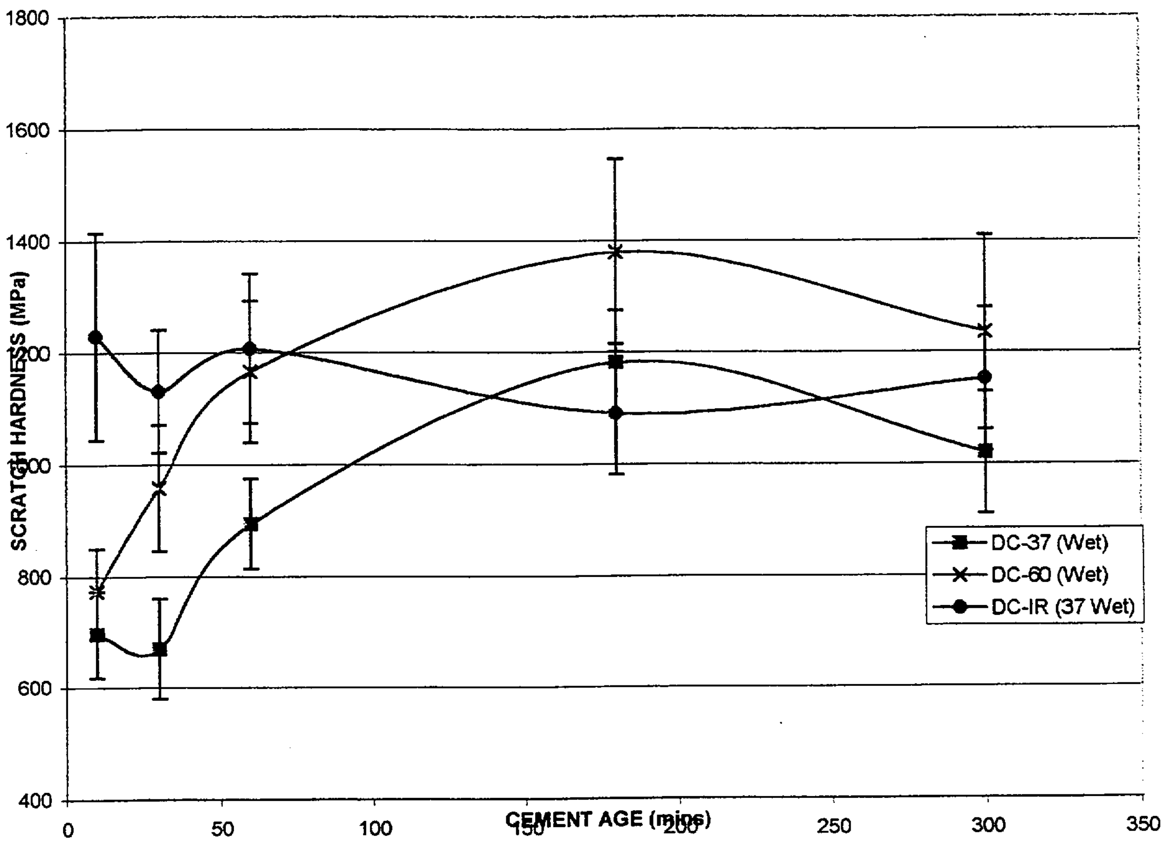


TABLE 7. THE MECHANICAL PROPERTIES OF KG23/5 CEMENTS WITH IR CURING

Cement age (mins)	Strength - IR	Hardness - IR	Strength - No IR	Hardness - No IR
0.25	44.44	0.0	10.8	337.9
0.5	34.69	0.0	31.94	641.8
1	62.32	0.0	64.05	924.5
3	99.06	0.0	94.03	1274.1
5	98.34	0.0	100.1	1351.6
24	115.2	1325.4	112.8	1339.5

TABLE 8. THE IR CURING OF GLASS-IONOMER CEMENTS

CEMENT AGE (mins)	Fuji II LC	KG23/5	KG23/5 - IR	Fuji II LC - IR
15	568.8	72.87	1010	1030
30	574.2	551	902	1050
60	659.9	730	852	1050
180	844.5	597	1120	1060
300	956	906	1120	1080
1440	1076.2	875	1230	1230

FIGURE 2. THE IR CURING OF GLASS-IONOMER CEMENTS

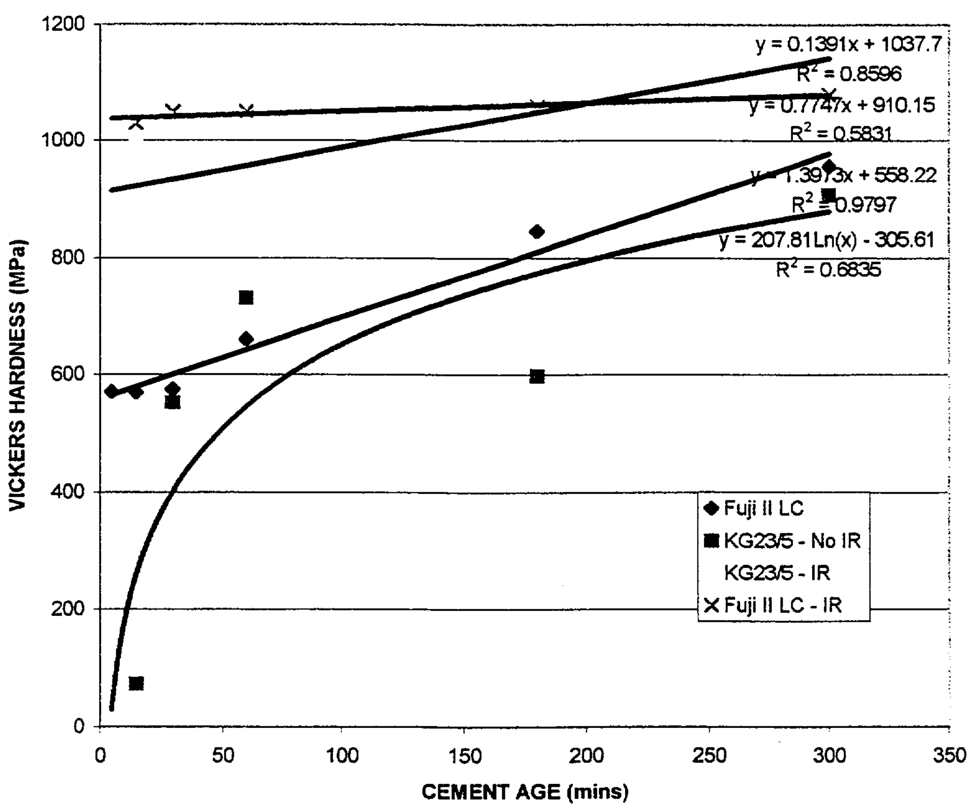


TABLE 9. THE 15-MINUTE HARDNESS VALUES OF KG23/5 CEMENT

Curing time (s)/	SCRATCH HARDNESS (MPa)		
Distance (cm)	20s	40s	60s
No IR		72	
1	684	956	1684
2	496	688	804
3	448	568	700
4	408	488	524
5	488	488	520
8	280	301	269

FIGURE 3. THE 15 MINUTE HARDNESS VALUES OF IR-CURED KG23/5 CEMENT

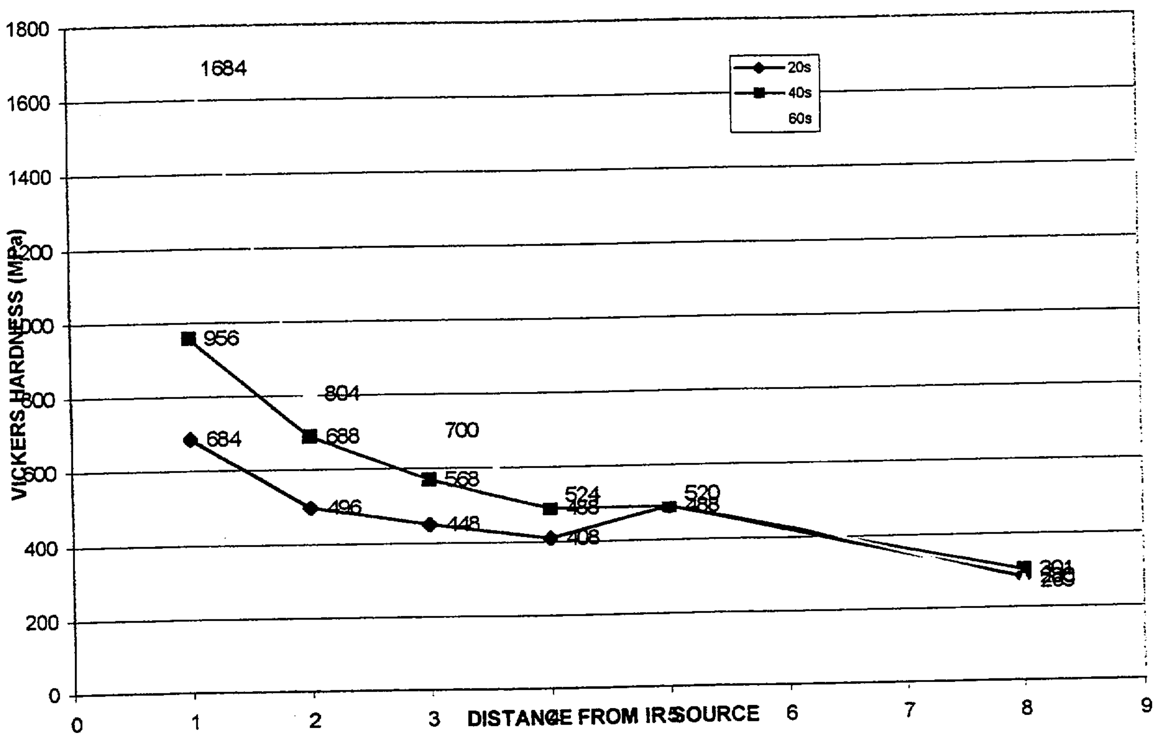
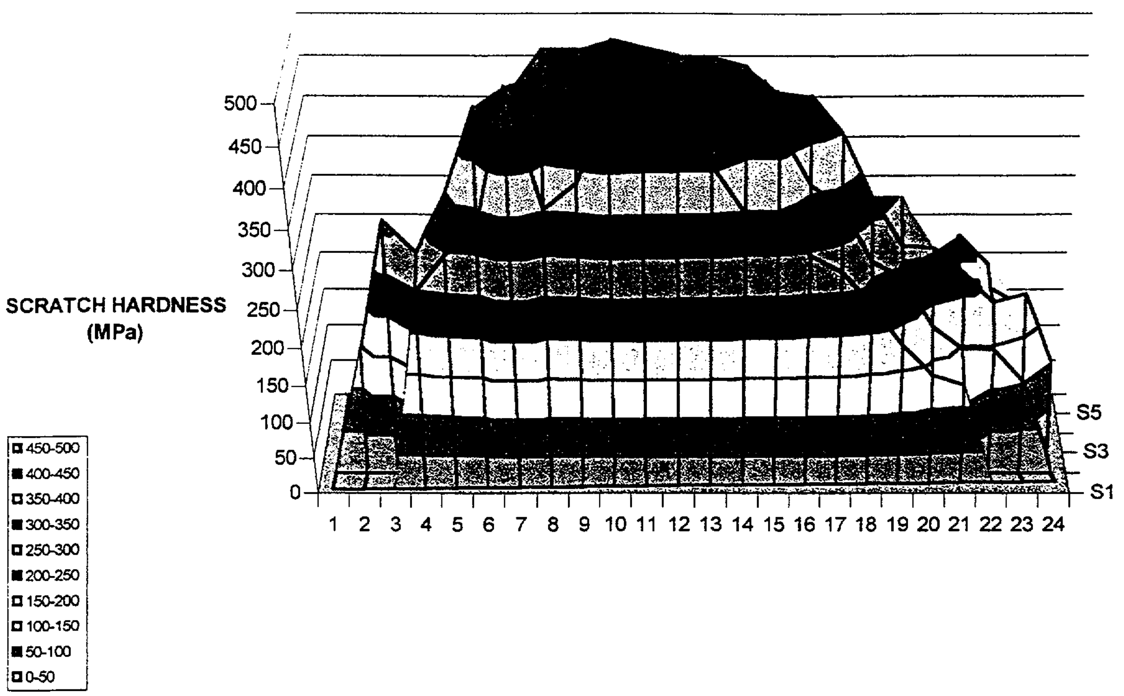


TABLE 10. THE CURING EFFICACY OF VARIOUS IR REGIMES

Cement Distance from IR (cm)	Hardness (0.01 value) %		
	60s	40s	20s
1	136.9	77.7	55.6
2	65.4	55.9	40.3
3	56.9	46.2	36.4
4	42.6	39.7	33.2
5	42.3	39.7	39.7
8	21.9	24.5	22.8

FIGURE 4. THE SURFACE HARDNESS PROFILE OF DC CEMENT CURED FOR 60s 1cm FROM THE IR ELEMENT



CLAIMS

1. The curing of acid-base cements, especially dental "ionomer" cements by infrared radiation (0.1 microns - 100 microns), but preferable 3 microns - 15 microns as a means of affecting, e.g. accelerating, their speed of set.

2. The curing of acid-base cements, especially dental "ionomer" cements by infrared radiation (0.1 microns - 100 microns), but preferable 3 microns - 15 microns as a means of accelerating their development of surface hardness.

3. The curing of acid-base cements, especially "ionomer" cements by infrared radiation (0.1 microns - 100 microns), but preferable 3 microns - 15 microns as a means of accelerating their resistance to water-contamination.

4. The above (1)-(3) improvements will produce "conventional" glass-ionomer cements capable of being polished immediately following IR curing. This is similar to the existing command curing of resin-modified ionomer cements by blue light

5. The IR curing of resin-based ionomer cements delivers the improvements (1)-(3), i.e. increase in speed of set, surface hardness and resistance to water solubility. These cements are already polishable following curing - (claim 4). The IR curing of non-resin ionomer cements delivers the properties (1)-(4)

6. The use of IR radiation as the sole source of external energy for the curing of ionomer cements, or the use of this radiation consecutively or simultaneously with any other energy source, e.g. blue light for all forms of compositions containing the "ionomer" reactions. At the present time, these include "conventional" acid-base glass-ionomer cements, resin-modified glass-ionomer cements, polyacid-modified composites (compomers) and all other blends or combinations of resins and ionomer components.

7. The device(s) capable of producing infrared radiation of the type indicated in Claim (1) and typified by our experimental IR unit or any modifications or versions intended or capable of curing ionomer cements by means including IR radiation. These include devices capable of producing IR radiation only or IR and other energy radiation, e.g. blue light simultaneously or consecutively.

Application No: GB 9901749.3
Claims searched: 1-7
Examiner: Dr Albert Mthupha
Date of search: 3 May 2000

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.R): C3V (VAM, VBC, VET)

Int Cl (Ed.7): A61K (6/00, 6/02, 6/083)

Other: CAS ONLINE, EPODOC, JAPIO, WPI.

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
X	Scand J Dent Res No. 92 (2), 1984, BRUNE et al, "Initial Acidity of Dental Cements", pages 156 to 160, especially pages 159-160.	1-7

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
		E	Patent document published on or after, but with priority date earlier than, the filing date of this application.
&	Member of the same patent family		