

REPLACED SHEETS		SG
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1 "Method of Deposition"

2

3 This invention relates to a method of depositing
4 substances, for example metals. The method may be used
5 in the manufacture of integrated circuits and
6 photomasks.

7

8 Modern technological demands in integrated circuitry,
9 for example opto-electronics and electronic surgical
10 implants, now require methods by which ultra fine metal
11 lines of submicron dimensions can be deposited onto
12 inert substrate materials. There is a considerable
13 demand for submicron technology in a wide variety of
14 disciplines, but there are many difficulties in
15 developing nanoscale metal deposition processes by
16 conventional lithographic methods. Lithography is the
17 process by which a pattern is transferred to the
18 surface of a substrate material. Before this process
19 can occur a photomask has to be prepared which defines
20 the pattern ultimately to be achieved on the substrate
21 layer.

22

23 Conventionally the photomasks required in the
24 manufacture of integrated circuits are made by applying
25 a film layer of metal, usually chromium since this

1 resists scratching or etching and has good adhesion
2 properties, by metal vapour deposition to a highly pure
3 quartz substrate which has been polished to give an
4 extremely flat surface. The exposed metal is then
5 covered with electron beam resist (which is a material
6 that is sensitive to a beam of electrons and is
7 disrupted on exposure thereto) by placing a drop of
8 resist in the middle of a spinning disk of mask
9 material. The resist is then baked and dried in an
10 oven. The usual electron beam resist is
11 polybutylsulphone (PBS).

12
13 An electron beam is directed at the resist face of the
14 resist/metal/substrate composite to degrade portions of
15 the resist, leaving unaffected a pattern required for
16 the integrated circuit. The degraded resist is then
17 dissolved away using a proprietary etchant to expose
18 the layer of metal in the areas where the resist has
19 been degraded, and this is subjected to wet etching by
20 ceric ammonium nitrate to remove the metal and expose
21 the quartz substrate. The remaining resist is then
22 burnt off from the residual metal, resulting in a
23 product through which light can pass apart from the
24 areas masked by metal.

25
26 The photomask thus produced is used to define a pattern
27 on a silicon chip coated with photoresist, by passing
28 ultraviolet light through the photomask to degrade the
29 photoresist in the defined areas.

30
31 This method of manufacturing photomasks has a number of
32 disadvantages. For example, the dissolution of the
33 degraded photoresist may not be entirely uniform, and
34 while the result is adequate for many products current
35 computer technology demands increasingly dense patterns

1 on integrated circuits and therefore higher-definition
2 photomasks, down to the nanoscale level.

3
4 A further area of loss of definition is the wet etch
5 process for removing the unwanted chromium. The etch
6 material attacks the upper, exposed face of the
7 chromium but as it penetrates into the chromium layer
8 its effect is not unidirectional, so instead of
9 producing vertical walls for the residual chromium it
10 erodes and undercuts these walls. This can produce a
11 scattering effect on the ultraviolet light during
12 manufacture of the integrated circuits, reducing
13 definition of the applied pattern. This is one of the
14 principal problems in mask-making.

15
16 Where several photomasks are used successively in the
17 manufacture of a chip, accurate registration of each
18 mask is absolutely essential so that each feature
19 appears in the correct place on the finished chip.
20 Problems in epitaxy can cause pattern shift and thus
21 registration. Usually registration is accurate to +/-
22 $0.2\mu\text{m}$. Registration problems, epitaxy growth problems,
23 undercutting, constructive and destructive interference
24 during lithographic process at micron and submicron
25 dimensions, all contrive to indicate that mask-making
26 at nanometer dimensions may require an entirely new
27 manufacturing process.

28
29 For nanoscale circuits, conventional methods using
30 metallic inks do not as yet possess the specifications
31 which can ensure the deposition of accurate straight
32 lines of high specification, and the formation of
33 perfect right angles rather than arcs during integrated
34 circuit manufacture. The low specification of the
35 conventional process of metallisation can result in

1 circuit overlap and circuit breakage which cannot be
2 tolerated when scribing nanoscale circuits. Further,
3 when ultra fine lines are required to carry current it
4 is a fundamental requirement that the circuit integrity
5 remains intact, and hence conductive materials which
6 are oxidized are not ideally suited to operate at such
7 dimensions.

8
9 According to the present invention there is provided a
10 method of chemical deposition comprising applying to a
11 substrate a compound which degrades under the effect of
12 a radiant or particle beam to produce a deposit,
13 applying to selected surface areas of said compound
14 such a radiant or particle beam and removing the
15 degraded compound and the unaffected compound from the
16 substrate. The deposit is left upon the substrate.

17
18 The process of the present invention causes degradation
19 of the compound only where contacted by the radiant or
20 particle beam. In other words, the dimensions of the
21 deposit caused by degradation of the compound is
22 proportional to the focal width of the irradiating
23 beam. Nanoscale dimensions may be achieved by the
24 process of the present invention.

25
26 Where the compound degrades to form a deposit of a
27 metallic or other conductive substance, then the method
28 may be used to manufacture integrated circuits directly
29 on the substrate. The deposit is preferably a metal or
30 metal alloy, and mention may be made of metals such as
31 gold, tin and chromium, or the deposit may be a
32 conductive non-metal or semi-metal, such as germanium.

33
34 In a further aspect there is provided a method of
35 manufacture of an integrated circuit, said method

1 comprising applying to a substrate a compound which
2 degrades under the effect of a radiant or particle beam
3 to produce a conductive, preferably metallic deposit,
4 applying to selected surface areas of said compound
5 such a radiant or particle beam and removing the
6 degraded compound and the unaffected compound from the
7 substrate.

8
9 Alternatively, the method of the present invention may
10 be used to manufacture a photomask which is then
11 subsequently used in lithography to manufacture
12 integrated circuits. In this embodiment the substrate
13 should be translucent or transparent and the deposit
14 formed by degradation of the compound should be opaque.
15 The deposit provides a barrier to passage of
16 ultraviolet light during use of the photomask in making
17 integrated circuits, as with the chromium in
18 conventional photomasks.

19
20 Thus in a yet further aspect, the present invention
21 provides a method of manufacture of a photomask, said
22 method comprising applying to a transparent or
23 translucent substrate a compound which degrades under
24 the effect of a radiant or particle beam to produce an
25 opaque deposit, applying to selected surface areas of
26 said compound such a radiant or particle beam and
27 removing the degraded compound and the unaffected
28 compound from the substrate.

29
30 In this embodiment the deposit formed by the action of
31 the particle or radiant beam need only be opaque and
32 there is no requirement for the deposit to be
33 conductive. Therefore the deposit may be of any
34 suitable opaque material, whether metal or non-metal.
35 The opaque deposit may be for example a metal or semi-

1 metal such as gold, tin, germanium or chromium, an
2 alloy, or a non-metal such as silicon or carbon.

3
4 In the present invention, the compound may be heat-
5 sensitive so that the use of a radiant beam in the form
6 of a laser beam degrades the compound to produce the
7 deposit.

8
9 Preferably, however, the particle beam is an electron
10 beam, in which case the compound may be for example an
11 organometallic compound which degrades under the effect
12 of the beam to deposit a metal on the substrate.

13
14 Conventionally the steric properties of the
15 organometallic material which may be used as the
16 compound in the process of invention are optimised by
17 selecting d^8 configurations which give square planar
18 geometry in the organometallic compound. Such
19 compounds include organometallic gold, platinum or
20 palladium fluorides.

21
22 In a yet further aspect, the present invention provides
23 a gold organometallic fluoride. Optionally the gold
24 organometallic fluoride may be used in the method of
25 deposition described herein.

26
27 Gold is an especially useful metal for inclusion in an
28 organometallic compound to be used in the method of
29 this invention, and a suitable organometallic form is
30 di-sec butylaurum (III) fluoride dimer.

31
32 Gold chemistry exists mainly in the Au(I) and Au(III)
33 oxidation states. Inorganic gold compounds usually
34 have the linear 2 co-ordination state and occasionally
35 exist in trigonal planar or tetrahedral configurations.

1 Au(III) compounds are in general square planar but can
 2 exist in five or six coordinated states. Simple
 3 organogold compounds (AuR - where R is any organic
 4 group) have not as yet been found although adduct
 5 compounds of Au(I) with phosphines or isocyanides are
 6 known. Trigonal planar Au(I) organoaurates (eg
 7 $\text{Li}[\text{AuCH}_3]_2$) exhibit low stability, spontaneously
 8 decomposing by reductive elimination of the methyl
 9 groups to give ethane. The trigonal planar gold
 10 complexes gain stability if complexed with larger
 11 ligands (eg $\text{Li}(\text{PMDT})[\text{AuCH}_3]_2$, where PMDT = pentamethyl-
 12 diethylenetrissamine)

13

14 Au(I) organyls are also accessible through carbene
 15 insertion into the Au-Cl bond.

16

17 eg $(\text{C}_2\text{H}_5)_3\text{PAuCl} + \text{CH}_2\text{N}_2 \text{ -----} \rightarrow (\text{C}_2\text{H}_5)_3\text{PAuCl} + \text{N}_2$

18

19 Au(III) organyls also require additional ligation to
 20 provide stability. Square planar compounds are
 21 obtained eg: $(\text{C}_6\text{F}_5)_2\text{AuClPPH}_3$. Halogen bridging Au
 22 complexes are known through the auration reaction:

23

24 eg: $2\text{Au}_2\text{Cl}_6 + 2\text{C}_6\text{H}_6 \text{ -----} \rightarrow \text{C}_6\text{H}_5\cdot\text{Cl}\cdot\text{Au}\cdot\text{Cl}\cdot\text{Cl}\cdot\text{Au}\cdot\text{Cl}\cdot\text{C}_6\text{H}_5$

25

26 Other typical organometallic compounds for use in this
 27 invention are polyalkyl metal fluorides, for example
 28 polydibutyltin difluoride, as these compounds contain
 29 generally linear chains which allow good definition of
 30 the affected areas of the compound on application of
 31 the radiant or particle beam.

32

33 The organometallic compounds are advantageously
 34 fluorides since the fluoride component enhances the
 35 adhesion between the substrate (such as silicon or

1 quartz) and the metal film formed after irradiation.
2 The enhanced adhesive is believed to be due to the
3 formation of Si-F bonds. This enhanced adhesion is not
4 achieved with the equivalent chloride and bromide
5 compounds. Particularly good adhesion is observed when
6 tetra-sec-butyldiaurum difluoride is used as the
7 organometallic compound. A further advantage is that an
8 especially even dispersion of organometallic compound
9 is observed where a fluoride compound is used.

10

11 Fluorine bridging in organogold complexes has not been
12 reported but pentafluorochalcogenate derivatives of
13 gold are known (ie $\text{Au}(\text{OTeF}_5)_3$). The group OTeF_5 is often
14 regarded as a pseudohalogen but more precisely is a
15 pseudofluorine. The ability of Au to form bridging
16 species renders the gold pentafluorochalcogenate
17 compound the only known binary transition metal
18 derivative of OTeF_5 . This compound is of a
19 centrosymmetric dimer with four terminal TeF_5 groups and
20 two μ -oxo bridging bidentate OTeF_5 groups

21

22 Further according to the invention there is provided a
23 method of preparing polydibutyltin difluoride,
24 comprising fluorinating polydibutyltin dichloride.

25

26 Polydibutyltin dichloride is commercially available but
27 is unstable at ambient temperature. The difluoride,
28 while known, has until now been very difficult to
29 produce.

30

31 The fluorination may be carried out using for example
32 sodium fluoride, and the resulting polydibutyltin
33 difluoride may be used in the manufacture of photomasks
34 by applying it, for example by spinning, to a
35 transparent or translucent substrate such as quartz.

1 In this process the quartz is rapidly rotated while the
2 polydibutyltin difluoride in solution is slowly
3 applied, for example by drip, to it. The solvent is
4 then driven off leaving an accurately-controlled layer
5 of the organometallic compound.

6
7 In some cases it is possible to deposit the opaque
8 material by application to the substrate of a solution
9 of the degradable compound and then driving off the
10 solvent, preferably by microwave heating which has the
11 effect of allowing the solvent to be removed from the
12 substrate interface early in the procedure, thereby
13 producing effective adhesion between the deposited
14 degradable compound and the substrate.

15
16 In investigating the yield of organometallic precursor
17 material extracted from the reactant mixture as a
18 function of solvent polarity, cyclohexane, n-hexane,
19 isopentane, chlorofluorocarbon 113 ($\text{CCl}_2\text{FCClF}_2$), CFC-
20 113a (CCl_3CF_3), n-pentane and iso-pentane have been
21 examined. The optimum solvent to provide a smooth even
22 distribution of organometallic material on a quartz
23 substrate was found to be a low-boiling-point aliphatic
24 hydrocarbon solvent. Hence, n-pentane or isopentane
25 are excellent solvents for work to be performed at
26 ambient temperatures.

27
28 Embodiments of the present invention will now be
29 described by way of illustration in the following
30 Examples.

31
32
33

1 Example 1

2

3 Preparation of Polydibutyltin difluoride

4

5 Analar grade methanol (50ml, B.D.H.) was transferred to
6 a conical flask containing 2.1g of dried sodium
7 fluoride (Aldrich Chemical Co.,). The flask was shaken
8 to dissolve the solid material. Dibutyltin dichloride
9 (0.5g, Aldrich Chemical Co.,) was added and the conical
10 flask was stoppered and sealed using paraffin film.
11 The system was shaken for 1 hour, then allowed to stand
12 for 12 hours at ambient temperature.

13

14 The reaction mixture was then passed through a sintered
15 glass filter to remove any undissolved sodium fluoride
16 prior to the eluent being transferred to a phase
17 separation vessel. Petroleum ether (bp. 60-80°C, 100
18 ml) was added to the eluent phase and the reaction
19 mixture shaken before allowing the mixture to settle
20 and separate. Using the upper phase, the purification
21 process was repeated a further three times using
22 methanol (100ml) before the upper phase was finally
23 transferred to a 250 ml round bottom flask. The flask
24 containing the reaction solution was affixed to a
25 rotary evaporator and allowed to evaporate down until a
26 precipitate appeared.

27

28 The solution was analysed by Raman Spectroscopy and
29 identified as a low molecular weight polymer of
30 dibutyltin difluoride.

31

32 Application and Preparation of Organometallic Overlayer

33

34 The solution of polydibutyltin difluoride monomer
35 prepared above was spun onto a clean polished quartz

1 plate. As the solvent evaporated off a film of
2 polymeric dibutyltin difluoride formed on the quartz
3 substrate. The organometallic overlayer supported on
4 the quartz substrate was placed in a JEOL T300 Scanning
5 Electron Microscope and bombarded with electrons
6 (energy 30 keV) for a time which varied from 5s to 10
7 min over an area of 100 x 100 μm . of the organometallic
8 sample, then moved to an adjacent area.

9
10 After irradiation of each area the sample was examined
11 using an optical microscope to reveal a linear track of
12 metallic particles, approx. 10 μm wide. The non-
13 degraded polydibutyltin difluoride film was removed by
14 dissolution in methanol.

15
16 Using the method of this embodiment it is possible to
17 produce a photomask of very high definition so that
18 dense and discrete patterns can be applied to silicon
19 chips to provide integrated circuits. The method
20 removes the need for affected electron beam resist to
21 be dissolved as in the conventional method, and also
22 for the wet etch process, both of which reduce the
23 definition and performance of the photomask.

24
25 Example 2

26
27 Preparation of Di-sec butylaurum (III) Fluoride dimer.

28
29 Magnesium turnings were degreased by washing in sodium
30 dried ether and transferred to a double-necked round
31 bottom flask containing a magnetic stirring bar.
32 Sufficient sodium dried ether was added to cover the
33 magnesium turnings. The flask was fitted with a water
34 cooled condenser and a dropping funnel charged with
35 5.62g dibutyl bromide (Aldrich Chemical Co.) in 80ml of

1 sodium dried ether. The butylbromide/ether mixture
2 was added slowly and allowed to react with stirring
3 with the magnesium turnings. The system was left to
4 react for 2 hours at ambient temperature.

5
6 The prepared butyl magnesium bromide/ether mixture was
7 decanted to a dry dropping vessel and fitted to a
8 double necked roundbottom flask containing an ethereal
9 solution of aurum (III) chloride (500mg in 20ml ether
10 Aldrich Chemical Co) cooled with powdered ice. The
11 ethereal solution of butyl magnesium bromide was added
12 slowly with stirring to the aurum (III) chloride
13 solution over a 20 minute period. After reaction
14 powdered ice was added to the reactant mixture which
15 was then allowed to warm up to ambient temperature.

16
17 The resultant organometallic gold halide was extracted
18 from the reactant mixture using isopentane in a phase
19 separation process. A dry methanol/isopentane phase
20 separation process was performed a further three times.
21 The product mixture was reacted with sodium fluoride
22 (Aldrich Chemical Co, 2.1g) dissolved in 50ml of dried
23 methanol (BDH), and shaken at ambient temperature over
24 a 24 hour period. The mixture was then passed through
25 a sintered glass filter to remove any undissolved NaF
26 prior to the eluent being transferred to a phase
27 separation vessel. Three phase separation cycles were
28 performed using an isopentane/dry methanol mixture
29 containing the reaction products, the hydrocarbon phase
30 being carried over in each step of the separations.
31 The final hydrocarbon phase was transferred to a round
32 bottom flask, affixed to a rotary evaporator, and
33 allowed to evaporate down until a precipitate appeared.
34 The product material had a white waxy appearance and
35 gave off a pungent odour. Contact of the product

1 material with the skin left a purple stain consistent
2 with the deposition of colloidal gold.

3
4 Characterisation of the product material was performed
5 using ^1H , ^{13}C , ^{19}F , and Distortionless Enhancement by
6 Polarization Transfer (DEPT) liquid phase NMR (nuclear
7 magnetic resonance) analysis using CDCl_3 or
8 deuterobenzene as solvent (Figures 1-5). Mass
9 spectroscopy was also used to identify the product
10 material (Figures 6-7).

11

12 Results:

13

14 ^1H NMR analysis of the product material gave peaks at
15 1.38 (hextet), 1.13 (hextet) and 0.79 (d of d) (Figures
16 1 and 1a). Two dimensional ^1H analysis of the material
17 shows three proton environments (Figure 2) and DEPT
18 analysis shows that the proton arrangement on the
19 organic liquid is one carbon containing an even number
20 of protons and three carbons containing an odd number
21 of protons (Figure 4). ^{13}C NMR analysis (Figure 3) of
22 the product material shows 4 peaks at 31.5, 29.7, 22.2,
23 11.7 ppm relative to TMS. The data indicates that four
24 carbon environments are present in the product
25 material. ^{19}F NMR analysis of the product material
26 (proton coupled and proton decoupled spectra) shows a
27 singlet peak at -66.3 ppm confirming the presence of
28 fluorine in the product. The ^{19}F NMR datum (Figure 5)
29 also indicates that the fluorine environment is
30 contained in a symmetrical field.

31

32 Mass spectroscopic analysis of the product material
33 confirms the molecular ion mass of the product material
34 to be 661 amu (Figures 6 and 7). Fragmentation of the
35 product material is consistent with the loss of the

1 following molecular fragments:-

2		
3	<u>Fragments</u>	<u>Residual Mass (amu)</u>
4		
5	CH ₃ - group	647
6		
7	2 @ CH-	632
8		
9	3 @ CH ₃ - + 1 @ C ₄ H ₉ -	558
10		
11	3 @ CH ₃ - + 1 @ C ₃ H ₇ - + 1 @ C ₄ H ₉ -	516
12		
13	1 @ Au	463
14		
15	1 @ Au + 1 @ C ₃ H ₇ -	421
16		
17	1 @ Au + 1 @ C ₃ H ₇ - + 1 @ F	402
18		
19	1 @ Au + 1 @ C ₄ H ₉ - + 1 @ F	385
20		
21	1 @ Au + 1 @ C ₄ H ₉ - + 2 @ F	368
22		
23	1 @ Au + 2 @ C ₄ H ₉ - + 1 @ C ₃ H ₇ -	
24	+ 2 @ F	269

25
26
27 UV analysis of an evaporated film of organoaurum
28 product supported on quartz shows that the material is
29 uv transparent in the range 900nm-350nm and hence the
30 material is suited for use in mask-making where uv
31 lithography is to be applied.

32
33 Studies have shown that the optimum solvent to be used,
34 both in the abstraction of the product material from
35 the phase separation stage of the preparation and in

1 the deposition of the material onto a quartz substrate,
2 is a low boiling point saturated alkane. Hence, the
3 product material was applied to the quartz substrate
4 using isopentane solvent.

5
6 The adhesion of organoaurum film to the quartz
7 substrate material is enhanced by exposing the coated
8 quartz substrate to microwave radiation prior to
9 electron beam bombardment. It is thought that this
10 process effectively evaporates off residual solvent -
11 trapped between the organoaurum film and the quartz
12 substrate to give better contact at the interface. The
13 quartz substrate can also be coated with the
14 organoaurum material via organometallic vapour
15 deposition technique (OMVD). The OMVD deposition
16 method results in a smoother more uniform coverage of
17 the organoaurum material compared to the solvent
18 application method.

19
20 Electron beam bombardment of the coated quartz
21 substrate was performed using a JEOL JSM-T220 Scanning
22 Microscope. The microscope was set at an accelerating
23 voltage of 20kV, focusing and movement of the sample
24 under the electron beam being both performed manually.
25 The organoaurum material easily reduced to the metal
26 component under the electron beam irradiation (Figure
27 8). The best line width attained to date using the
28 focussing power available on the JEOL instrument is
29 500nm.

30
31 A sample of the dibutylaurum (III) bromide dimer was
32 applied to the quartz substrate material and subjected
33 to electron beam bombardment. A film of reduced gold
34 metal was obtained on the quartz substrate.

35

1 Modifications and improvements can be made without
2 departing from the scope of the invention, for example
3 by using vapour phase deposition of the metal-
4 containing polymer.

5
6
7 Example 3

8
9 Preparation of Dibutylplatinum (IV) Fluoride dimer.

10
11 Magnesium turnings were degreased by washing in sodium
12 dried ether and transferred to a double-necked round
13 bottom flask containing a magnetic stirring bar.
14 Sodium dried ether was added sufficient to cover the
15 magnesium turnings. The flask was fitted with a water
16 cooled condenser and a dropping funnel charged with
17 5.62g dibutyl bromide (Aldrich Chemical Co.) in 80ml of
18 sodium dried ether. The butylbromide/ ether mixture
19 was added slowly and allowed to react with stirring
20 with the magnesium turnings. The system was left to
21 react for 2 hours at ambient temperature.

22
23 The prepared butylmagnesium bromide/ether mixture was
24 decanted to a dry dropping vessel and fitted to a
25 double necked round bottom flask containing 500mg of
26 platinum (IV) chloride (Aldrich Chemical Co) cooled
27 with powdered ice. The butylmagnesium bromide mixture
28 was added slowly with stirring to the platinum (IV)
29 chloride solution. After reaction powdered ice was
30 added to the reactant mixture and allowed to warm up to
31 ambient temperature.

32
33 The dibutylplatinum bromide dimer was extracted from
34 the reactant mixture using n-pentane in a phase
35 separation process. A dry methanol/ n-pentane phase

1 separation process was performed a further three times.
2 The product mixture was reacted with sodium fluoride in
3 dry methanol as described for the polydibutyltin
4 difluoride process.

5
6 The dibutylplatinum (IV) fluoride dimer product was
7 applied to a clean quartz substrate from an n-pentane
8 solution and n-pentane was driven off in a microwave
9 cooker to bake the product onto the quartz. The
10 microwave process ensured that the n-pentane was driven
11 off from the quartz interface instead of from its own
12 surface. This gave very good deposition of the product
13 on the quartz and allowed very good definition after
14 electron beam bombardment. A film of reduced platinum
15 metal was obtained supported on the quartz substrate.

16
17 A sample of the dibutylplatinum (IV) bromide dimer was
18 applied to the quartz substrate material and subjected
19 to electron beam bombardment. A film of reduced
20 platinum metal was obtained on the quartz substrate.

21
22 Examples 4-7

23
24 Preparation of Propyl, Tertiary butyl,
25 cyclohexylmethyl, benzyl analogues

26
27 An analogous procedure to that described in Example 2
28 was performed to prepare the propyl, tertiary butyl,
29 cyclohexylmethyl and benzyl analogues of organo gold
30 halides. The preparation of the organo magnesium
31 halide intermediate involved reaction of degreased
32 magnesium turnings with the ethereal solutions of the
33 appropriate propyl, tertiary butyl, cyclohexylmethyl
34 and benzyl bromides respectively.

35

1 Results:

2
3 a) NMR analysis of the Propyl Auric Halide Material:

4
5 The ^1H NMR of the product material from the
6 reaction of the propyl magnesium Grignard with an
7 ethereal solution of auric (III) chloride is shown
8 in figure 9. The NMR shows peaks at 0.89 ppm
9 (triplet), and 1.29 ppm (multiplet) relative to
10 TMS (tetramethylsilane), which is consistent with
11 the spectrum expected from the propyl ligand. The
12 broad band at 1.57 ppm is consistent with that
13 expected for the $-\text{CH}_2-$ adjacent to a group inducing
14 electron shielding. The ^{13}C NMR is shown in Figure
15 10. Although noisy, the spectrum shows three
16 peaks at 14 ppm, 22 ppm and 33 ppm relative to
17 TMS. The ^{13}C environments are consistent with CH_3- ,
18 $-\text{CH}_2-$ and a shielded $-\text{CH}_2-$ respectively. The ^{19}F
19 NMR of the sample (proton coupled and decoupled)
20 showed a singlet at -67.64 ppm relative to CCl_3F
21 indicating that the material contained fluorine in
22 a symmetrical field.

23
24 The product material was applied to a quartz
25 substrate as described in Example 2. Electron
26 beam bombardment was applied to the coated quartz
27 material as described in Example 2. The material
28 reduced under the electron beam bombardment to
29 give gold lines identical to those obtained from
30 the tetrabutyl diaurum difluoride material.

31
32 b) NMR analysis of the Benzyl Auric Halide Material:

33
34 The ^1H NMR spectrum of the product material is
35 presented in figure 11. The NMR data shows major

1 peaks at 7.3 ppm (multiplet) and 2.9 ppm (singlet)
2 relative to TMS. Smaller peaks consistent with
3 trace quantities of isopentane are also evident.
4 The spectrum shows that the product material
5 contains ligated benzyl groups. The ^{13}C spectrum
6 is presented in figure 12. The spectrum shows
7 peaks at 141.7 ppm, 128.5 ppm, 128.3 ppm, 125.9
8 ppm and 37.9 ppm relative to TMS. The ^{13}C
9 environments are consistent with the distribution
10 of shielding effect over the benzyl group ligated
11 to an electron inducing species. The ^{19}F NMR
12 showed a singlet at -67 ppm relative to CFC-11,
13 similar to those obtained for the previous
14 samples.

15
16 The product material from the benzyl Grinard
17 reagent reaction with an ethereal solution of auric
18 (III) chloride was applied to a clean quartz
19 substrate and subjected to electron beam
20 bombardment as described in Example 2. The
21 material did not reduce to give metal lines.

22
23 c) NMR analysis of the t-Butyl Auric Halide material.

24
25 The ^1H NMR analysis of the product material from
26 the reaction of the t-butyl Grinard reagent with
27 an ethereal solution of auric (III) halide is
28 presented in figure 13. The NMR data shows the
29 presence of a major peak at 1.24 ppm (singlet)
30 relative to TMS which is consistent with that
31 expected for the t-butyl group. Impurities in the
32 NMR have been identified to be MeOH, TMS and CHCl_3 .
33 Peaks consistent with some trace hydrocarbon
34 solvent are also observed. The ^{19}F NMR analysis
35 showed one peak at -67 ppm relative to CFC-11.

The product material from the reaction was applied to a clean quartz substrate and subjected to electron beam bombardment as described in Example 2. The material did not reduce to give gold metal lines.

d) NMR analysis of the Cyclohexylmethyl Auric (III) Material:

The ^{13}C NMR spectrum is presented in figure 14. The spectrum shows peaks at 36.15 ppm, 34.67 ppm, 33.6 ppm and a doublet at 26.6 ppm relative to TMS. The spectrum is consistent with that expected for methyl cyclohexane. The ^1H NMR analysis is presented in figure 15. The spectrum shows peaks at 1.2 ppm, (singlet) 1.6 and 1.7 ppm (singlets), 2.9 and 3.25 ppm (doublets). The spectrum is consistent with that of methylcyclohexane. The ^{19}F spectrum is shown in figure 16. A singlet peak at -68 ppm relative to CFC-11 is again observed.

The product material from the methylcyclohexyl aurum reaction was applied to a clean quartz substrate and subjected to electron beam bombardment as described in Example 2. The material did reduce under the conditions used to deposit fine lines of gold as observed under optical magnification. The width of the gold lines obtained were ca 500nm.

Example 8

Preparation of organoplatinum halide complex:

1 The preparation of platinum analogue materials was
2 performed analogously to those described in Example 2.
3 Propyl, butyl and methylcyclohexane analogues were
4 prepared. The respective organomagnesium halide
5 intermediate material was reacted with an ethereal
6 solution of platinum (IV) chloride (Johnson Matthey)
7 0°C. Phase separation of the product material was
8 performed as described in Example 2. The product
9 material obtained had a white waxy appearance similar
10 to that obtained from the gold complexes.

11

12

13 Results

14

15 a) NMR analysis of the product material from t-Butyl
16 platinum fluoride:

17

18 The ¹H NMR analysis is presented in figure 17. The
19 NMR shows the major peak at 1.25 ppm (singlet)
20 with evidence of MeOH solvent in the sample. The
21 ¹⁹F NMR analysis gave one peak at -67.66 ppm.

22

23 b) NMR analysis of the Butyl platonic halide product.

24

25 The ¹H NMR of the product obtained from the
26 reaction of the n-butyl Grignard reagent with an
27 ethereal solution of platinum (II) chloride is
28 presented in figure 18. The NMR shows peaks at
29 0.82 ppm (doublet), 1.28 ppm (singlet), 1.39 ppm
30 (singlet) and 1.5 ppm (singlet), relative to TMS.
31 The spectrum is consistent with that expected for
32 an n-butyl moiety. The ¹⁹F NMR analysis of the
33 material showed a singlet peak at -67 ppm (Figure
34 19).

35

The product material was applied to a clean quartz substrate and subjected to electron beam bombardment as described in Example 2. The material was readily reduced under the influence of the electron beam to deposit a line of metallic appearance.

c) NMR Analysis of the Propyl Platinic Fluoride Material:

The ^1H NMR spectrum was performed in deuterobenzene solvent and is presented in figure 20. The spectrum shows peaks at 1.33 ppm (singlet), 0.91ppm (singlet) and 0.39 ppm (singlet) relative to TMS. The spectrum is consistent with that expected for a propyl moiety. ^{19}F NMR analysis gave a single peak at -67 ppm.

d) NMR Analysis of the Methylcyclohexyl Platinic Fluoride Material:

The ^1H NMR spectrum of the product material from the reaction of the methylcyclohexyl Grignard reagent with an ethereal solution of platinum (II) chloride is presented in figure 21. The spectrum is consistent with that expected for the methylcyclohexyl ligand. The ^{13}C carbon NMR is also presented in figure 22. The spectrum also confirms that the product material contains ligated methylcyclohexyl groups.

Example 9

Example of the Butylpalladium analogue complex:

1 The preparation of the butyl palladium analogue was
2 performed as described in Example 2. The respective
3 organomagnesium halide intermediate material was
4 reacted with an ethereal solution of palladium (II)
5 chloride. It was observed that the product material
6 was thermally unstable resulting in deposition of the
7 palladium component at around 50°C. The deposited
8 palladium metal had strongly adhered to the glass
9 surface and could only be removed using acids.

11 Results

13 The ^1H NMR analysis of the product material from the
14 butyl Grignard reagent with an ethereal solution of
15 palladium (II) chloride is presented in figure 23. The
16 spectrum is consistent with that expected for a butyl
17 ligand a weak interaction with the ligated centre. The
18 ^{13}C NMR spectra shows the presence of 3 carbon
19 environments in the compound. The ^{19}F NMR again shows a
20 singlet peak at -67 ppm (Figure 25).

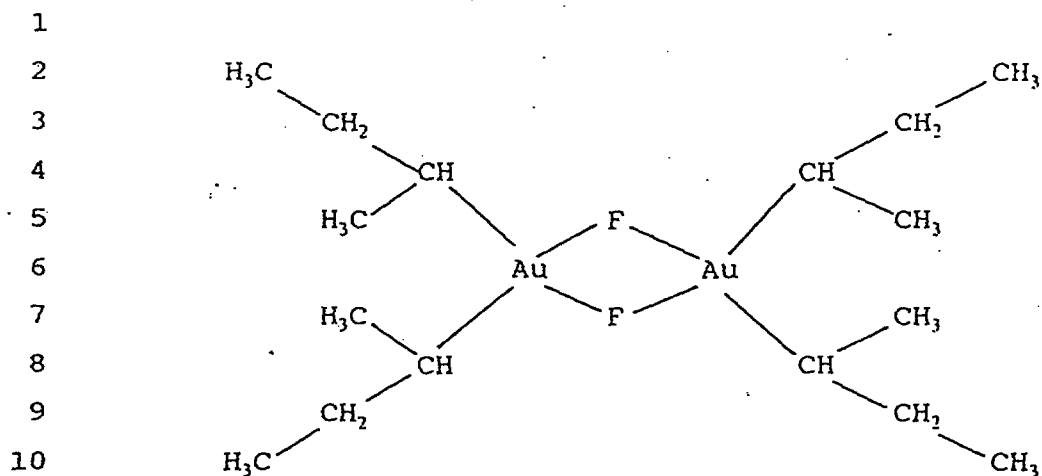
23 General Discussion

25 Examples 2 to 9 show that the butyl-, propyl- and
26 cyclohexyl- gold complexes are able to be reduced to
27 the metal component under electron beam bombardment.
28 The preparation of the propyl- and cyclohexyl-
29 analogues of the gold complex show no rearrangement
30 during the synthesis treatment. The ^1H NMR spectra of
31 these compounds do not exhibit any splitting patterns
32 that are consistent with the incorporation of fluorine
33 into the organic ligand. Whether these compounds have
34 fluorine as a constituent in their structure remains to
35 be determined. The ^{19}F NMR signal for the product

1 materials indicate that fluorine is present in a form
2 which confers a symmetrical field around the fluorine
3 environment. Given that fluorine has the ability to
4 affect neighbouring carbon atoms and cause coupling
5 over long ranges, it seems that whatever form the
6 fluorine is in, it is shielded from the organic ligands
7 so that coupling cannot take place.

8
9 The reaction of the n-butyl group to give sec-butyl
10 products gives rise to some questions about the
11 chemistry that is affecting the formation of the
12 butylauric (III) fluoride. Firstly, it is important to
13 note that the organic ligand itself does not contain
14 fluorine. No splitting of the proton signals are
15 observed in the proton spectra and there are no signals
16 in the ^{19}F NMR to indicate that hydrofluoroalkane groups
17 are present. Hence, the isomerisation step of the n-
18 alkane cannot be taking place during the fluorination
19 process. Isomerisation usually requires the production
20 of a carbocation species in the presence of F^- ions
21 during the reaction with the methanol solution of NaF
22 would immediately give rise to a fluorinated organic
23 ligand, for which there is no evidence. Hence, the
24 evidence suggests an intramolecular re-arrangement at
25 some stage in the production of the fluorided material.

26
27 The mass spectroscopic data gives a molecular ion of
28 661 amu, the fragmentation pattern of which is wholly
29 consistent with the formula $(\text{sec-butyl})_4 \text{Au}_2 \text{F}_2$. It must
30 be remembered that the Au (III) is in a $5d^8$
31 configuration that would give a square planar
32 arrangement, and would also consider the propensity of
33 gold to form bridging dimers. Thus, it is tentatively
34 submitted that the tetrakis sec-butyl aurum (III)
35 difluoride structure would be as follows:



12 The organoaurum product is uv transparent in the region
13 from 900-3500nm. This is a positive result should mask-
14 making be an application for the material.

15
16 No deposition of metal lines was obtained from the
17 benzyl- and tertiary butyl- analogues. These materials
18 possess good inductive properties and are able to pump
19 electron density into neighbouring moieties. This may
20 have an effect on the reducibility of the gold
21 complexes.

22
23 Electron reduction has been successfully shown on the
24 butyl analogue of the Pt material. Also the cyclohexyl
25 analogue of the Pd materials were able to be reduced by
26 electron beam bombardment techniques. Thermal
27 degradation of the palladium complex at 50°C resulted in
28 a strongly bound metallic film of palladium that could
29 only be removed by reaction with acid. This result
30 indicates that the palladium analogue of the
31 organometallic complexes is a suitable candidate for
32 metal deposition by laser degradation.



1 CLAIMS

- 2
- 3
- 4 1. A method of chemical deposition comprising
- 5 applying to a substrate a compound which degrades
- 6 under the effect of a radiant or particle beam to
- 7 produce a deposit and a degraded compound residue,
- 8 applying to selected areas of said compound a
- 9 radiant or particle beam and removing the degraded
- 10 compound residue and unaffected compound from said
- 11 substrate.
- 12
- 13 2. A method as claimed in Claim 1 for manufacturing
- 14 an integrated circuit wherein said deposit is a
- 15 conductive material.
- 16
- 17 3. A method as claimed in Claim 1 for manufacturing a
- 18 photomask wherein said substrate is a transparent
- 19 or translucent material and wherein said deposit
- 20 is opaque.
- 21
- 22 4. A method as claimed in any one of Claims 1 to 3
- 23 wherein said compound is an organometallic
- 24 material.
- 25
- 26 5. A method as claimed in any one of Claims 1 to 4
- 27 wherein said compound is an organometallic
- 28 fluoride.
- 29
- 30 6. A method as claimed in any one of Claims 1 to 5
- 31 wherein said compound is an organometallic gold,
- 32 platinum, palladium or tin fluoride.
- 33
- 34 7. A method as claimed in any one of Claims 1 to 6
- 35 wherein said compound is tetra-sec butyl diaurum



- 1 difluoride.
- 2
- 3 8. A method as claimed in any one of Claims 1 to 7
- 4 wherein said compound is exposed to microwave
- 5 radiation after application to said substrate and
- 6 before application of said radiant or particle
- 7 beam.
- 8
- 9 9. A method as claimed in any one of Claims 1 to 8
- 10 wherein said compound is applied to said substrate
- 11 by an organometallic vapour deposition (OMVD)
- 12 technique.
- 13
- 14 10. A method as claimed in any one of Claims 1 to 9
- 15 wherein said compound is degraded by a laser beam,
- 16 an ultra-violet beam and/or an electron beam.
- 17
- 18 11. An integrated circuit obtainable by the method as
- 19 claimed in any one of Claims 1, 2 and 4 to 10.
- 20
- 21 12. A photomask obtainable by the method as claimed in
- 22 any one of Claims 1 and 3 to 10.
- 23
- 24 13. An integrated circuit or photomask as claimed in
- 25 either of Claims 11 and 12 having nanoscale
- 26 resolution.
- 27
- 28 14. An integrated circuit manufactured by use of a
- 29 photomask as claimed in either of Claims 12 and
- 30 13.
- 31
- 32 15. A gold organometallic fluoride compound.
- 33
- 34 16. Use of an organometallic compound in a method as
- 35 claimed in any one of Claims 1 to 10.



- 1 17. Use as claimed in Claim 16 wherein said
2 organometallic compound is an organometallic gold,
3 platinum, palladium or tin fluoride compound.
4
- 5 18. Use of a radiant or particle beam in the method as
6 claimed in any one of Claims 1 to 10.
7
- 8 19. A method of manufacturing an organometallic
9 fluoride compound suitable for use in the method
10 of Claims 5 to 10, wherein sodium fluoride is
11 added to said organometallic compound in its
12 chloride or bromide form.
13
14

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1 ABSTRACT

2

3 METHOD OF DEPOSITION

4

5

6 There is described a method of chemically depositing a
7 substance. The method is of utility in the direct
8 manufacture of integrated circuits and in the
9 manufacture of a photomask for use in production of
10 integrated circuits. The method involves the use of a
11 compound which degrades into a deposit and a residue
12 when a radiant beam (eg a laser beam) or a particle
13 beam (eg an electron beam) is applied. The residue and
14 any unreacted compound may be washed off the substrate
15 to which it has been applied. Nanoscale dimensions of
16 the deposit can be achieved. A particularly suitable
17 organometallic compound is tetra-sec butyl diaurum
18 difluoride.

19

20

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25

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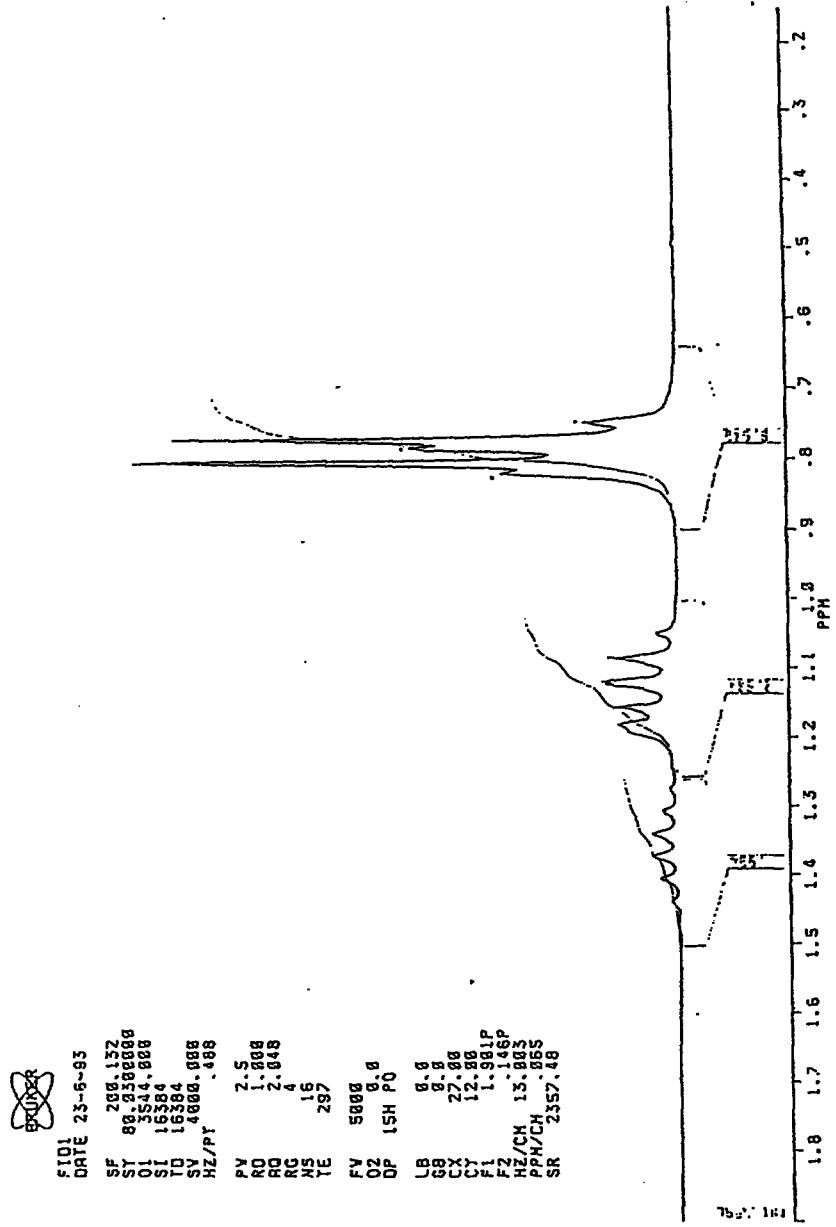
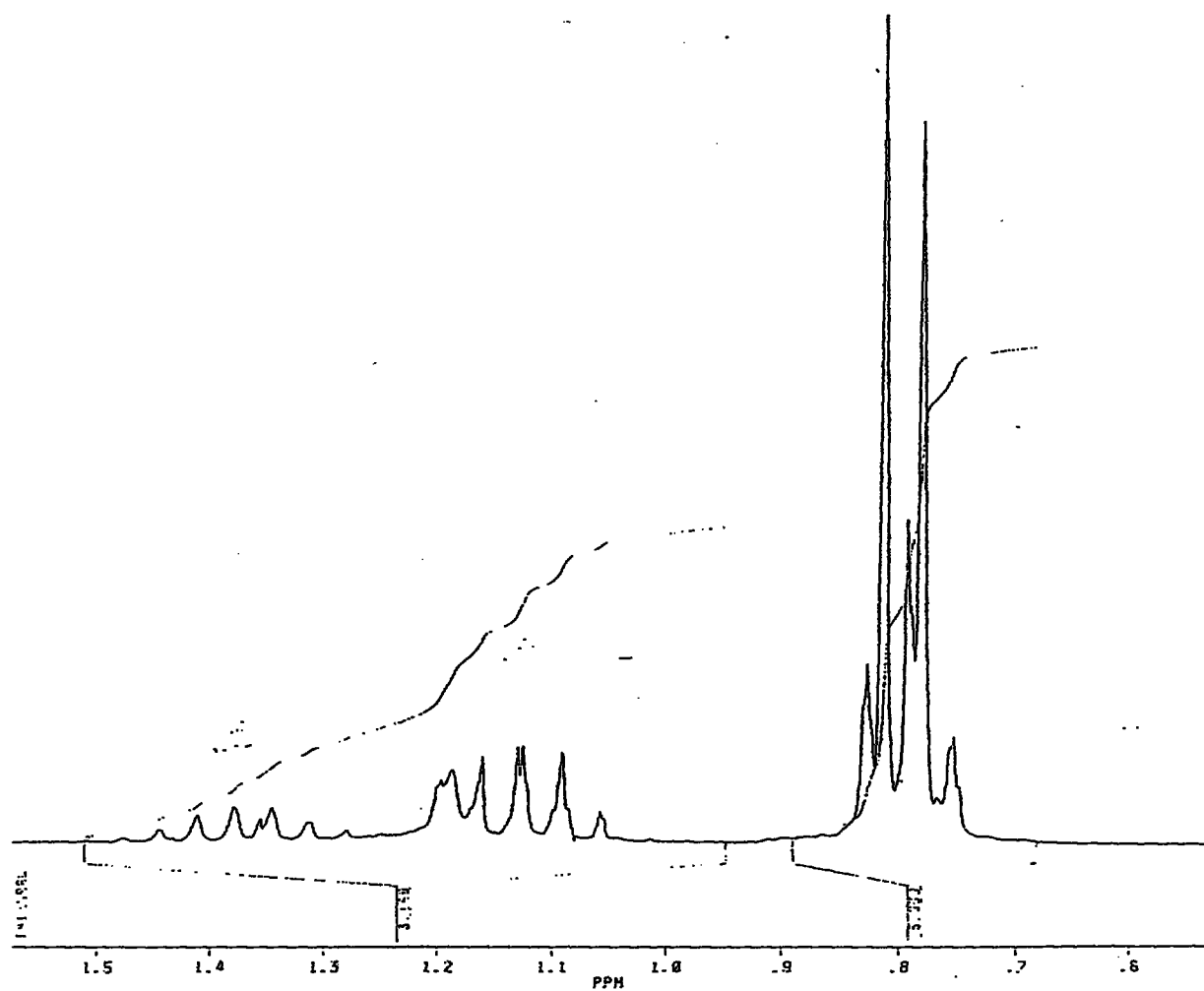


Fig 1

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226-019

268.248

2023-09-14

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767-632

232-928
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子孫一昌

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Abstract

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Index

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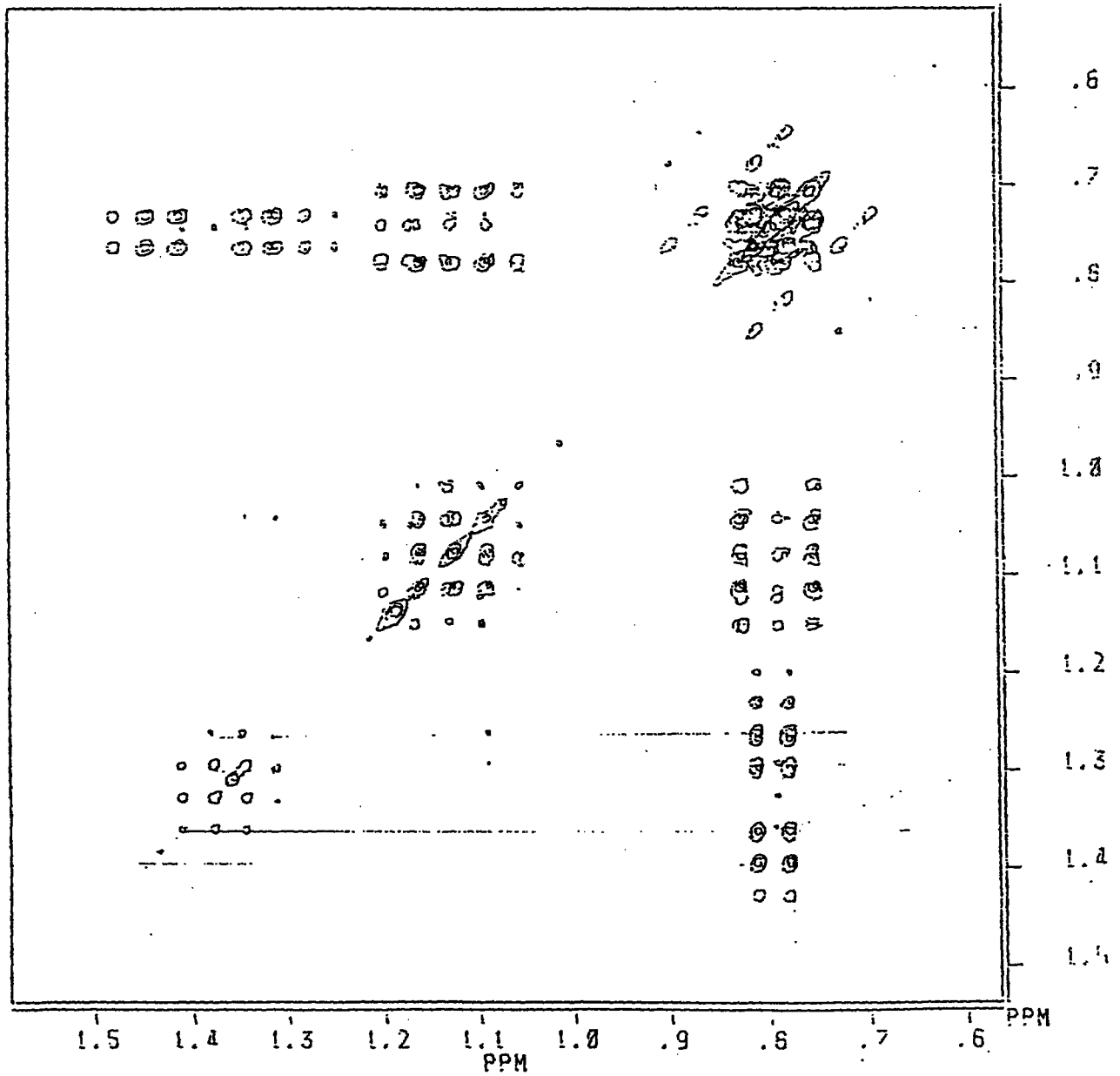
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Abstract

Fig 1a

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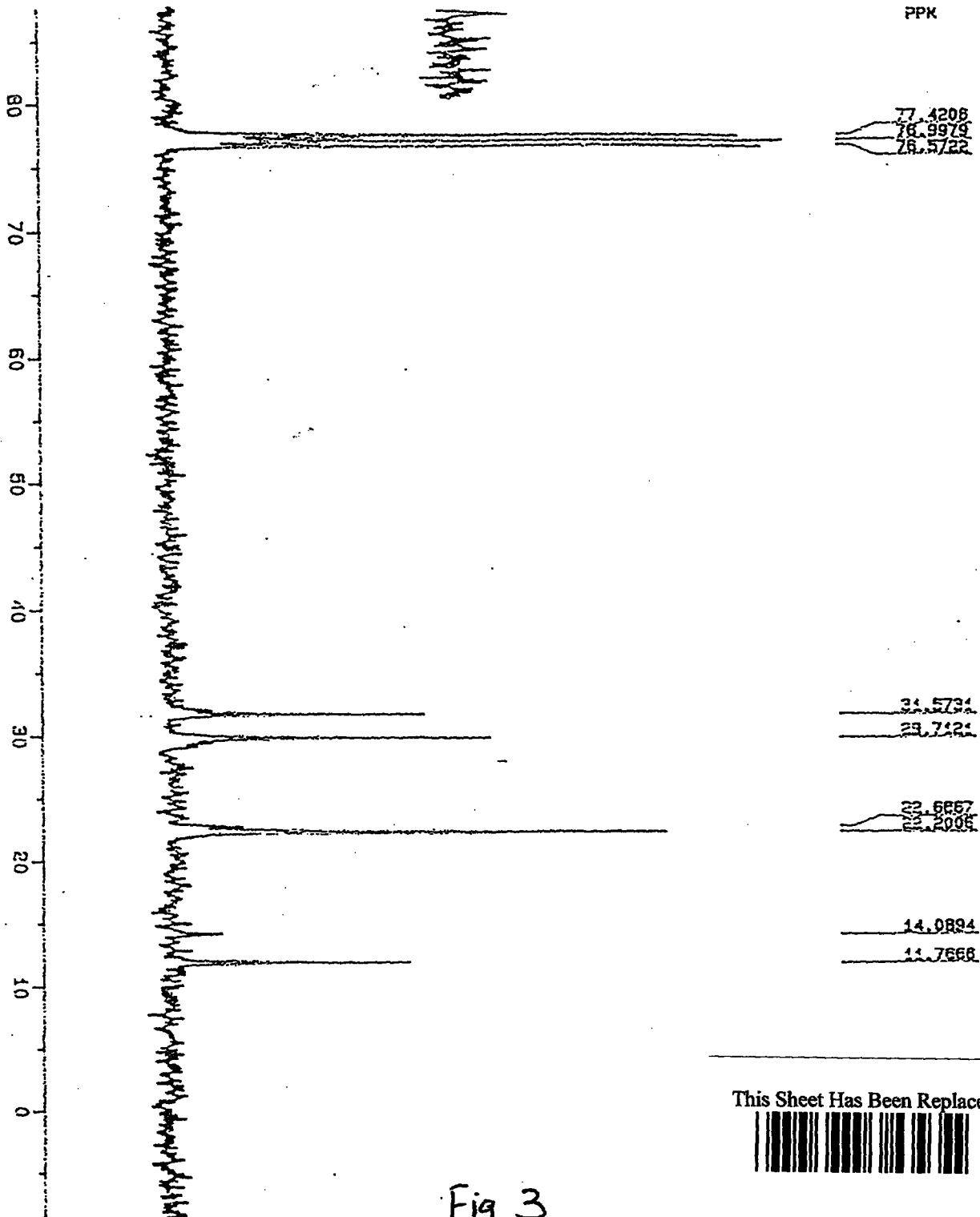
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Fig 2

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PPK



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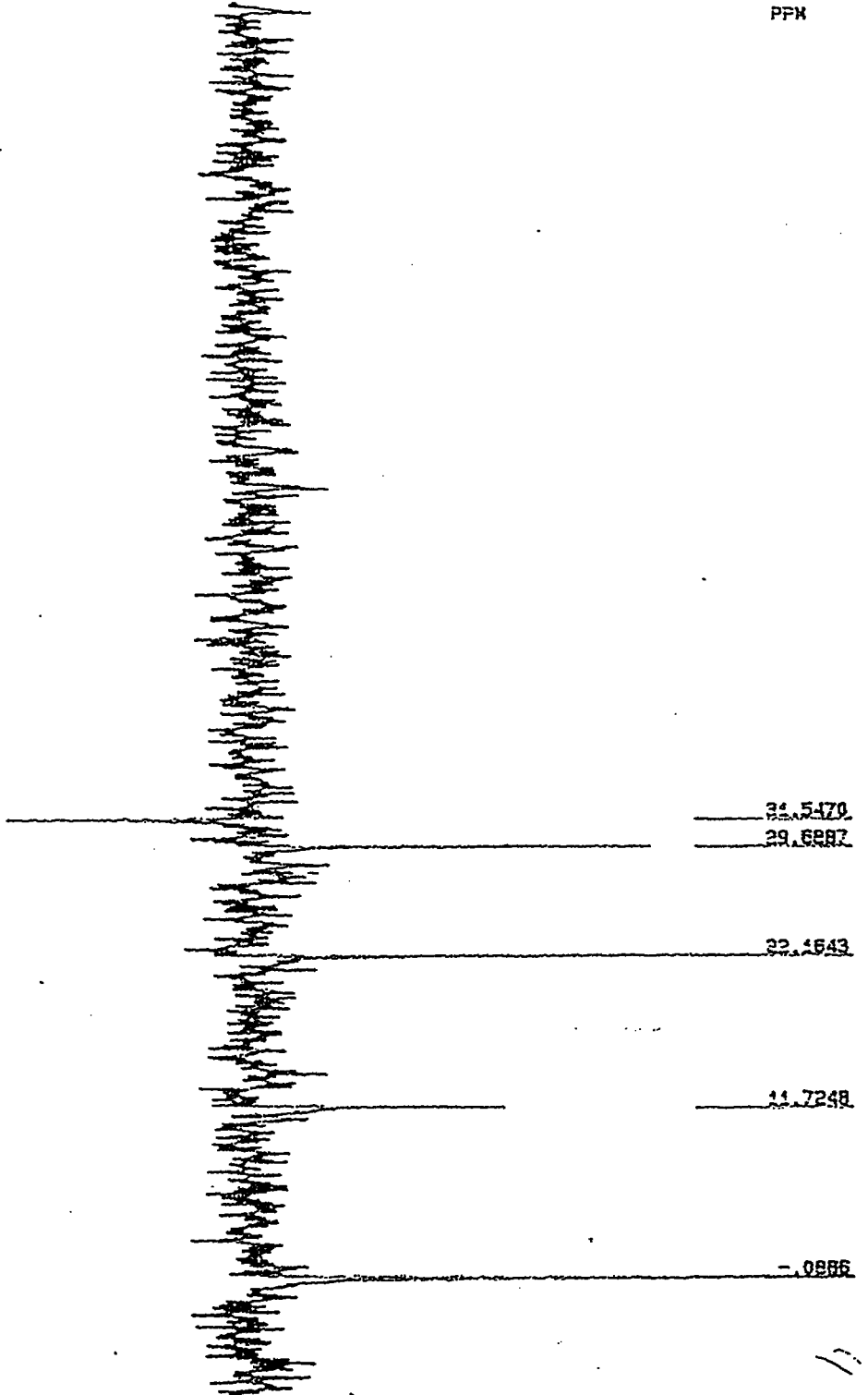
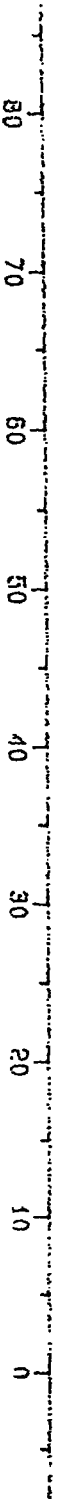
Fig 3

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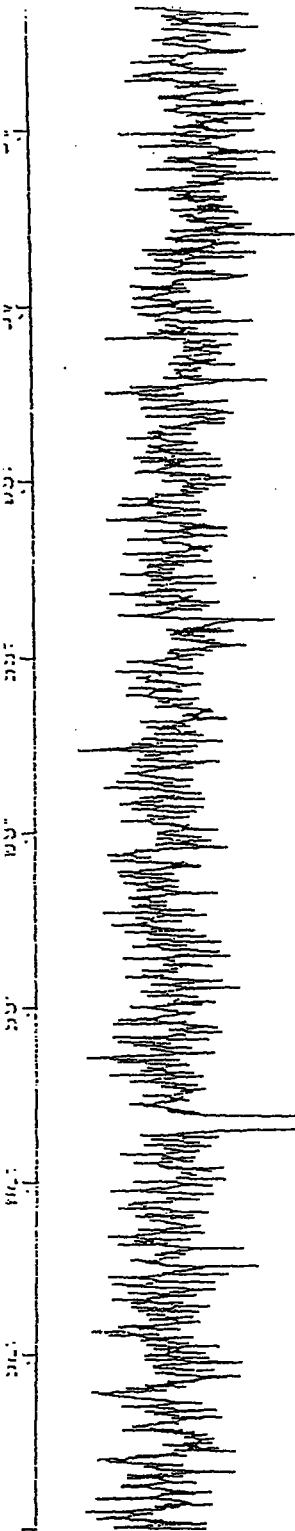


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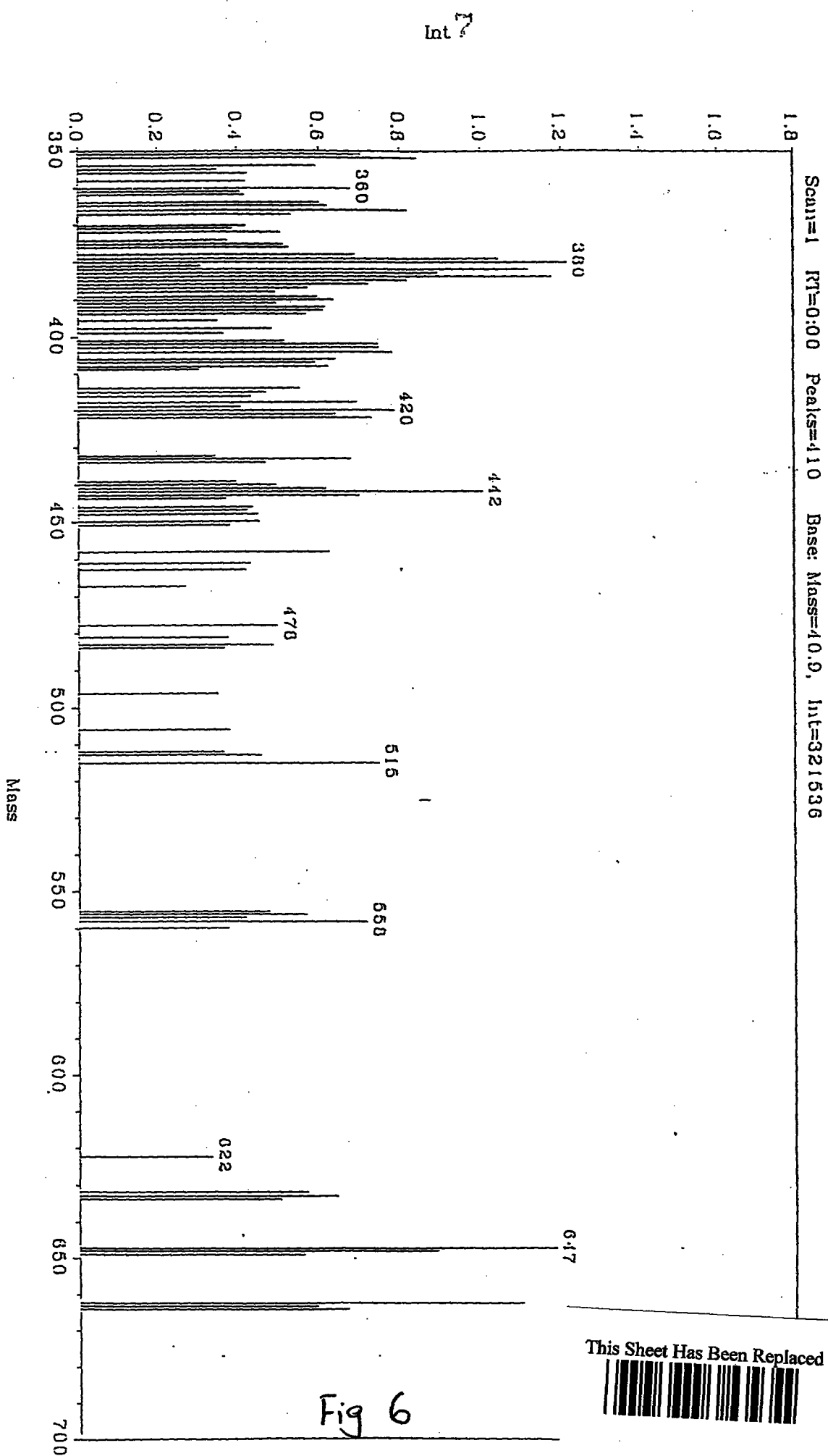
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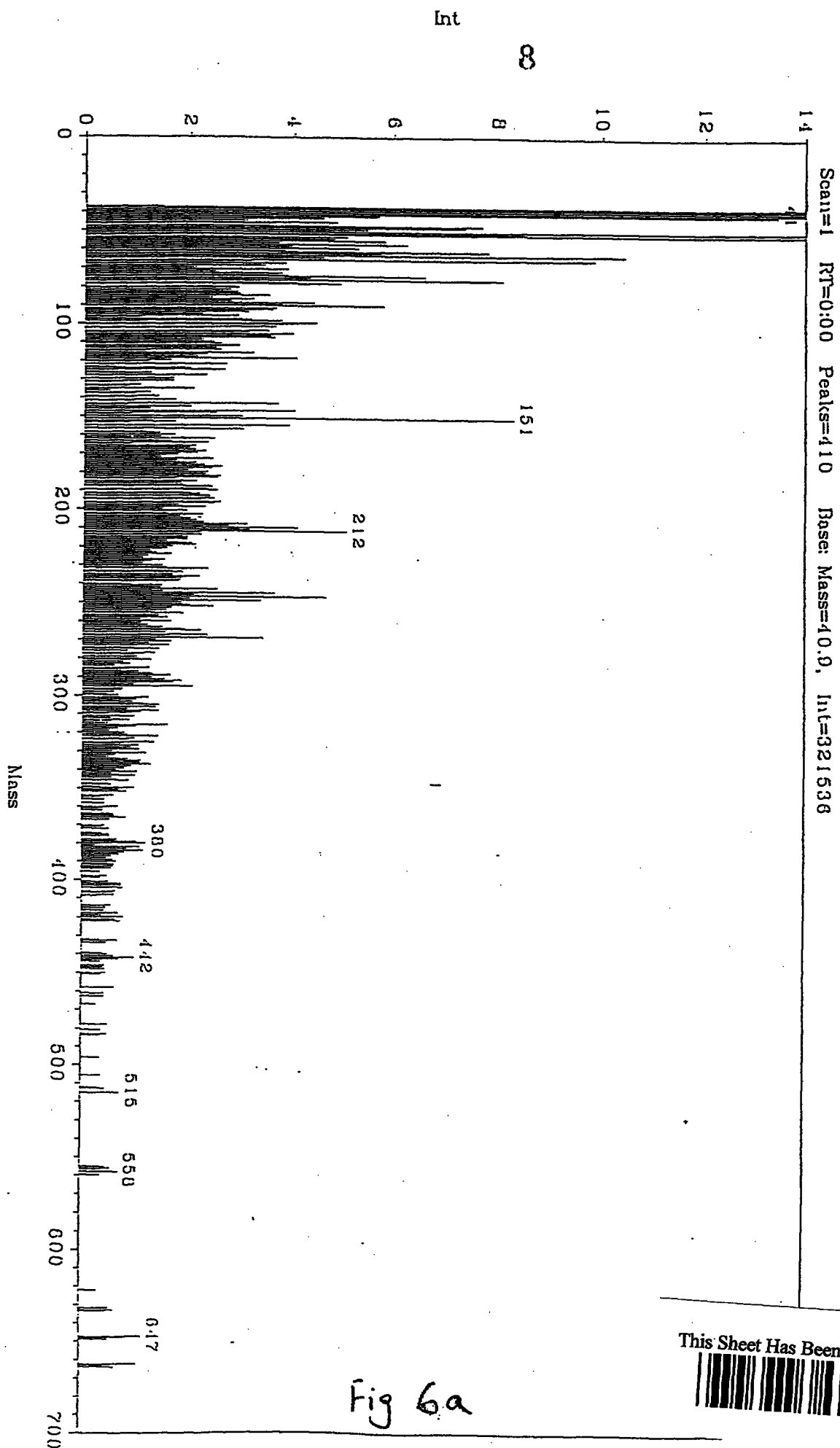


Fig 6a

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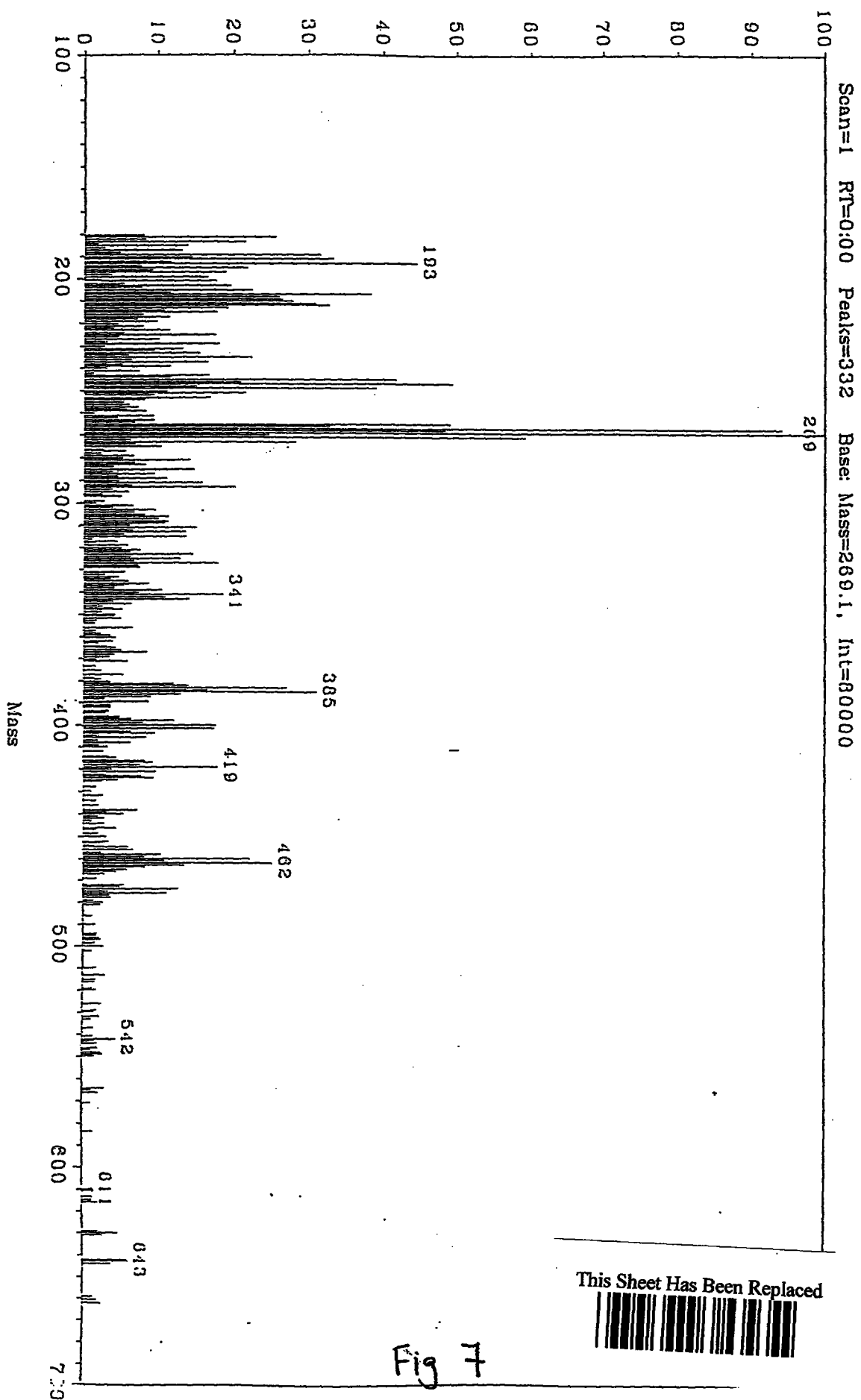
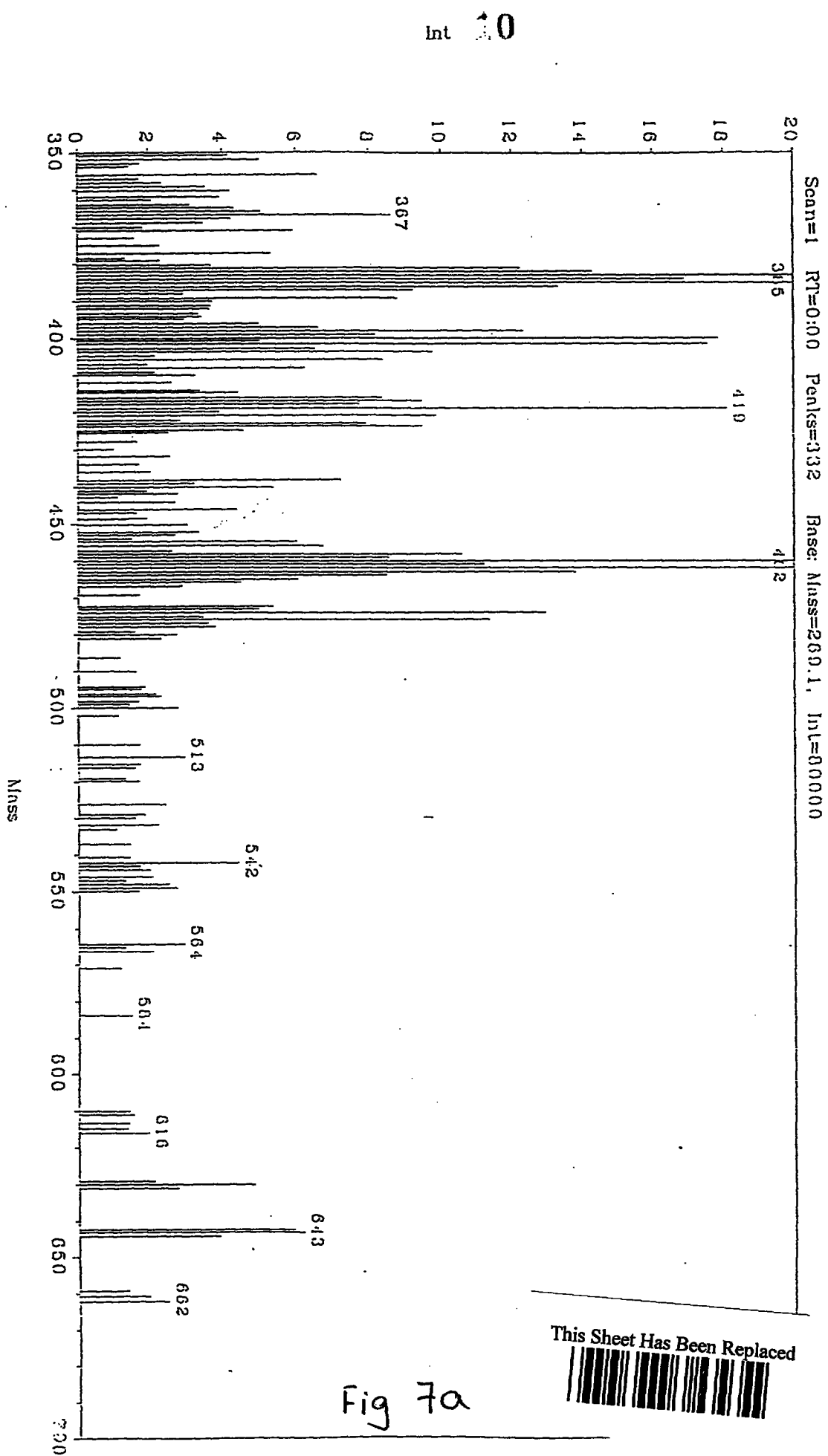


Fig 7

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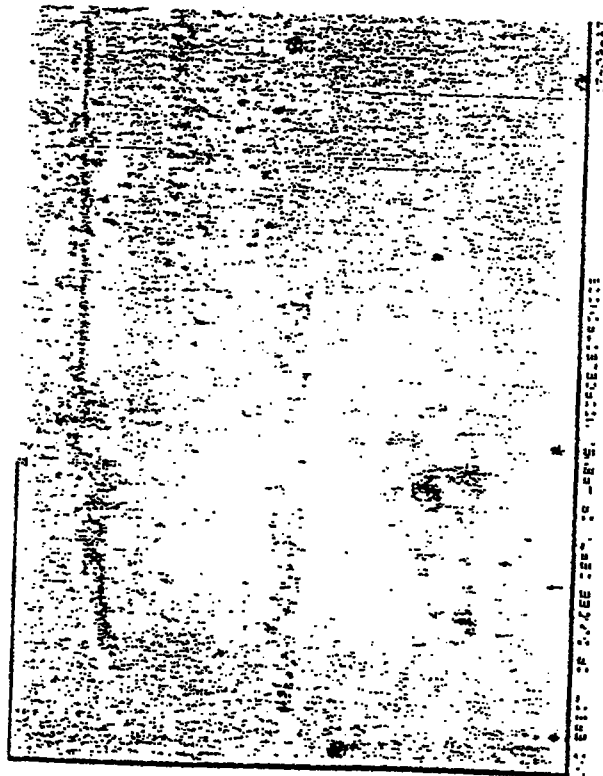
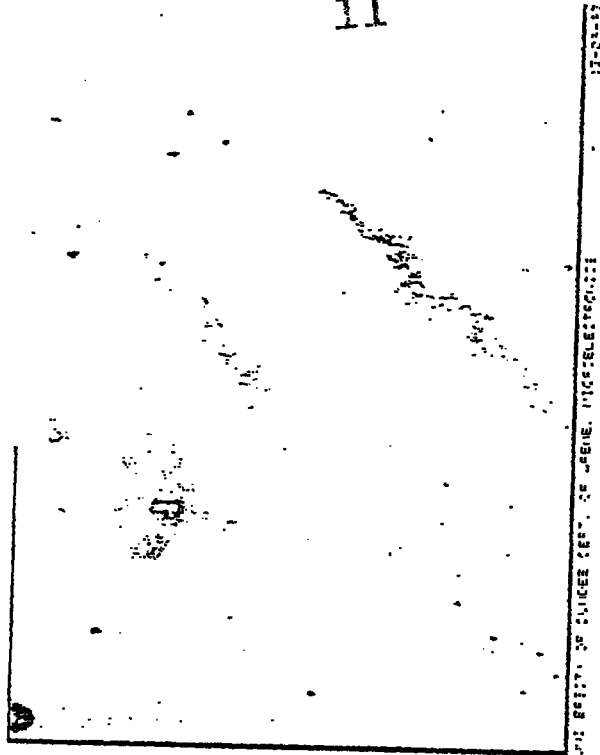


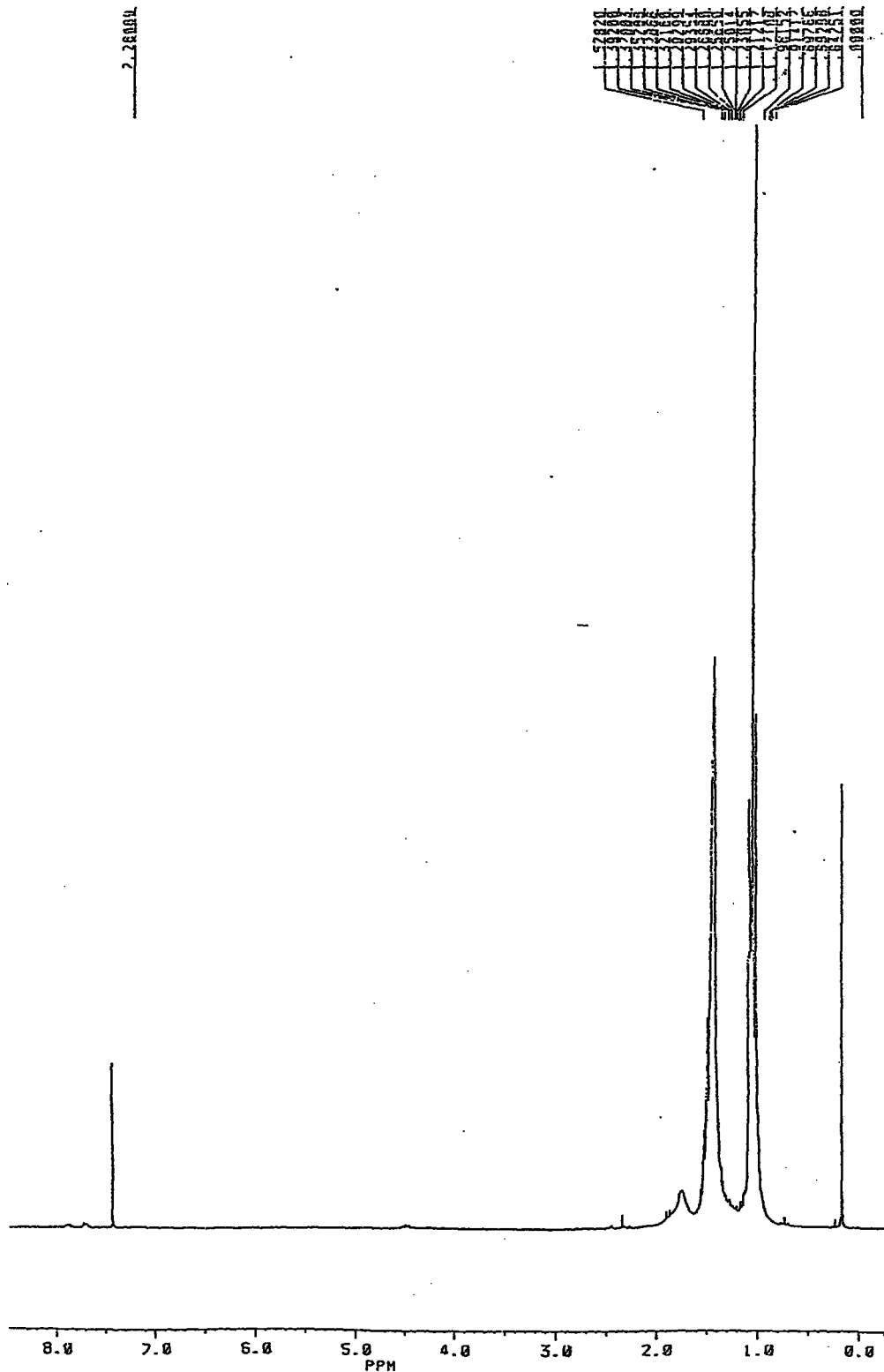
Fig 8

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FV 5000
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Fig 9

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PROPYL GOLD FLUORIDE

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38.1410
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EX-102

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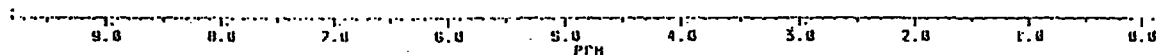
Fig 10

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14

Sample 11



NU-F

~~UNRES~~

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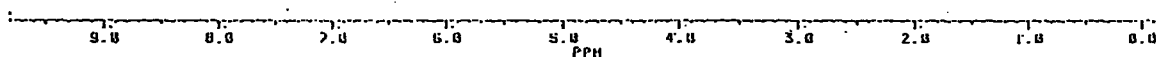
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11



NU-F

HERTZ

112081.1

Fig 11

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Tert Butyl Au F

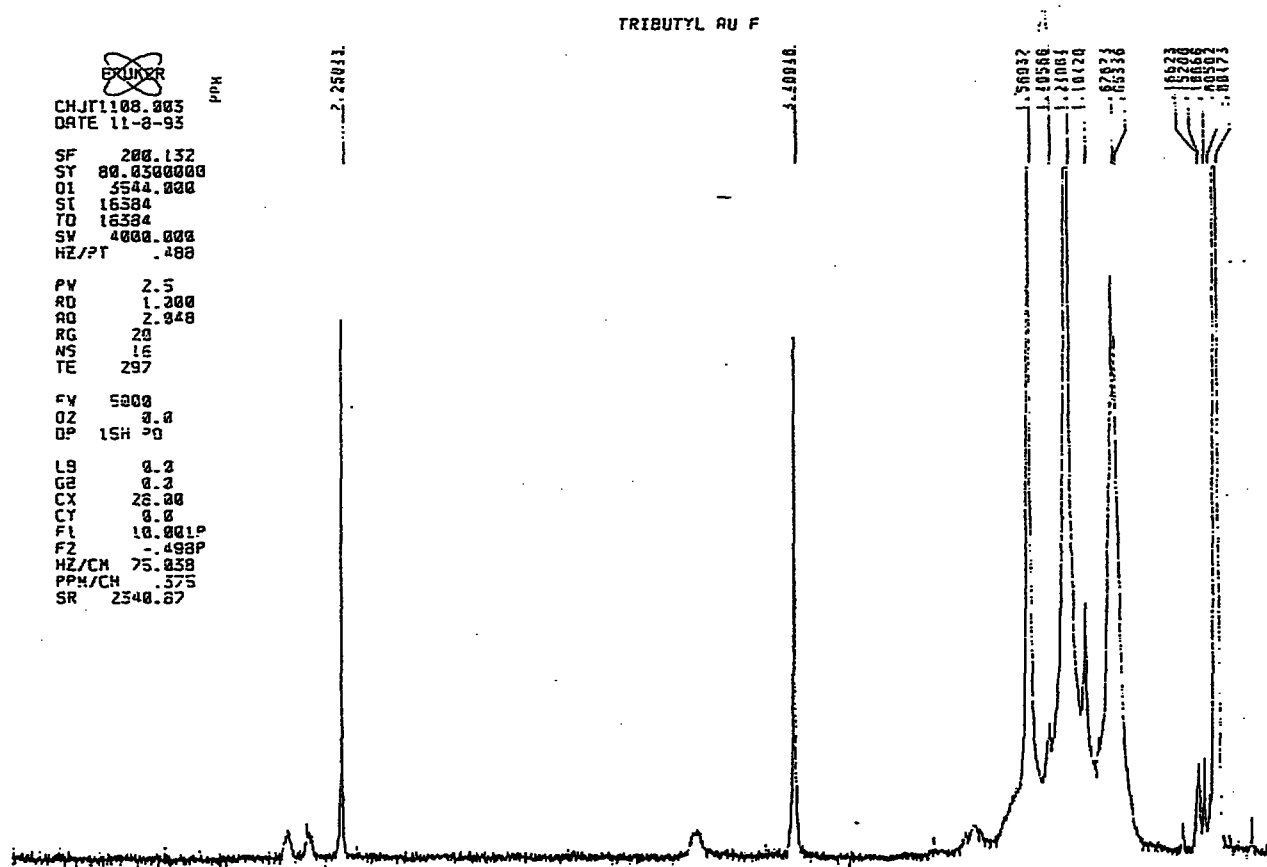


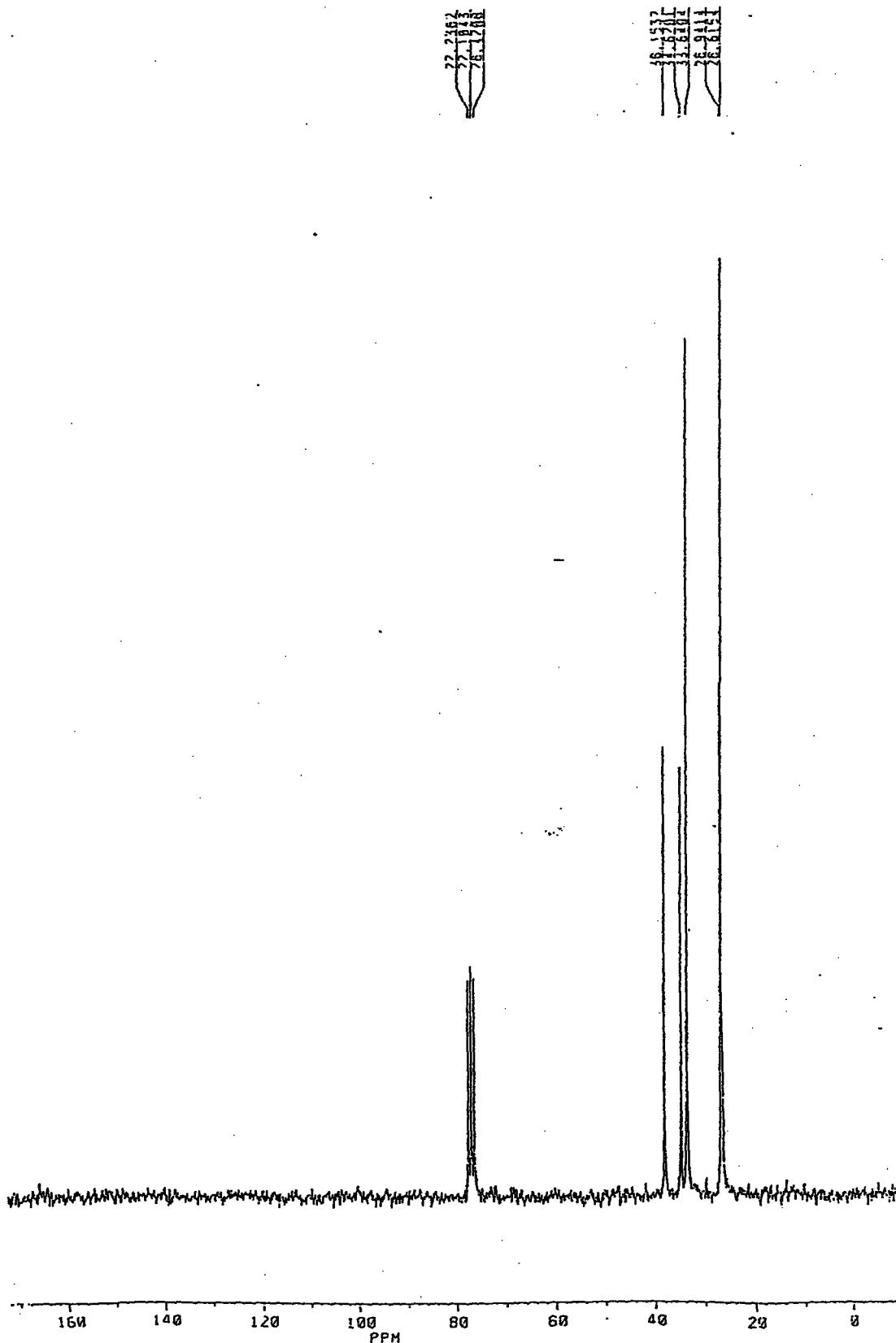
Fig 13

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17

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Fig 14

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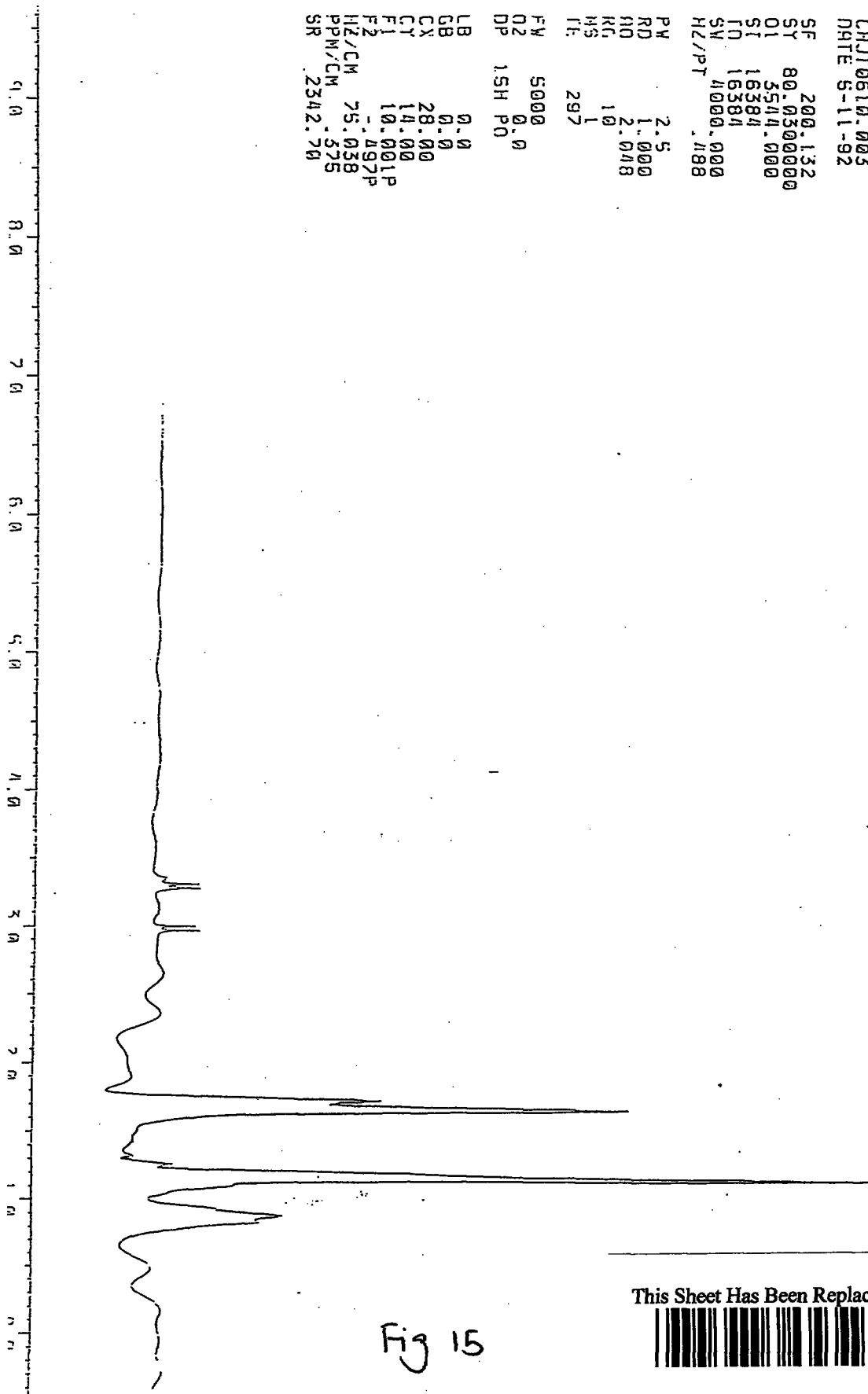
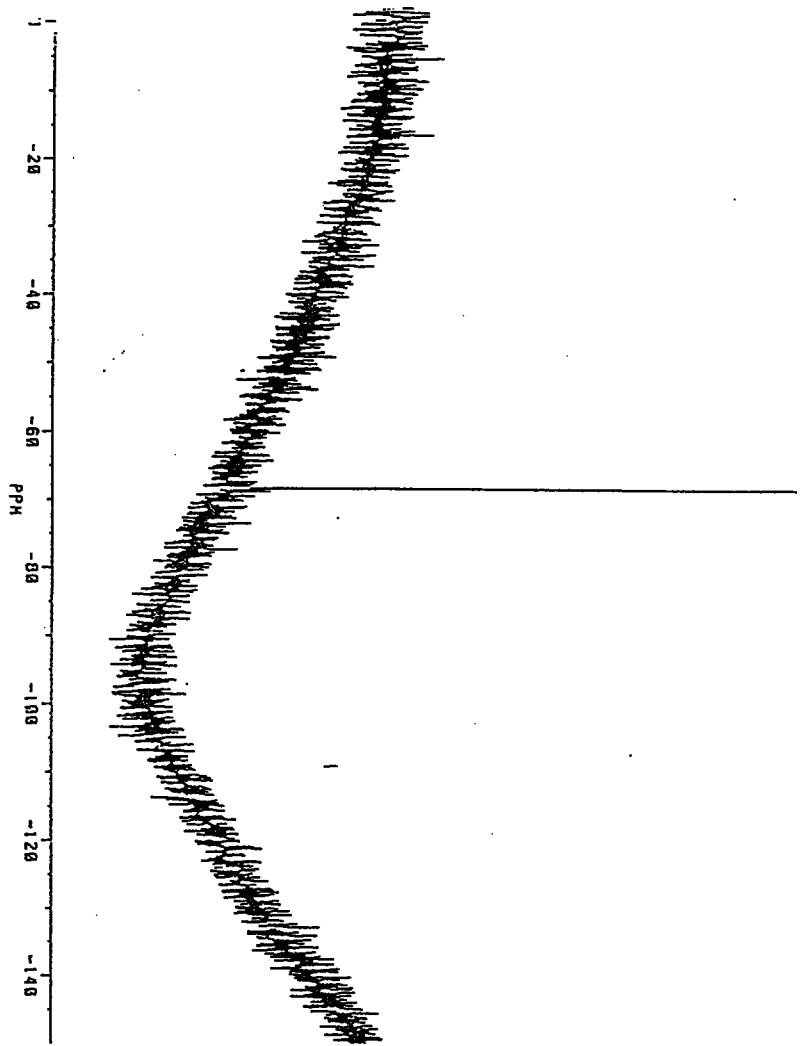


Fig 15

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Fig 16

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¹F
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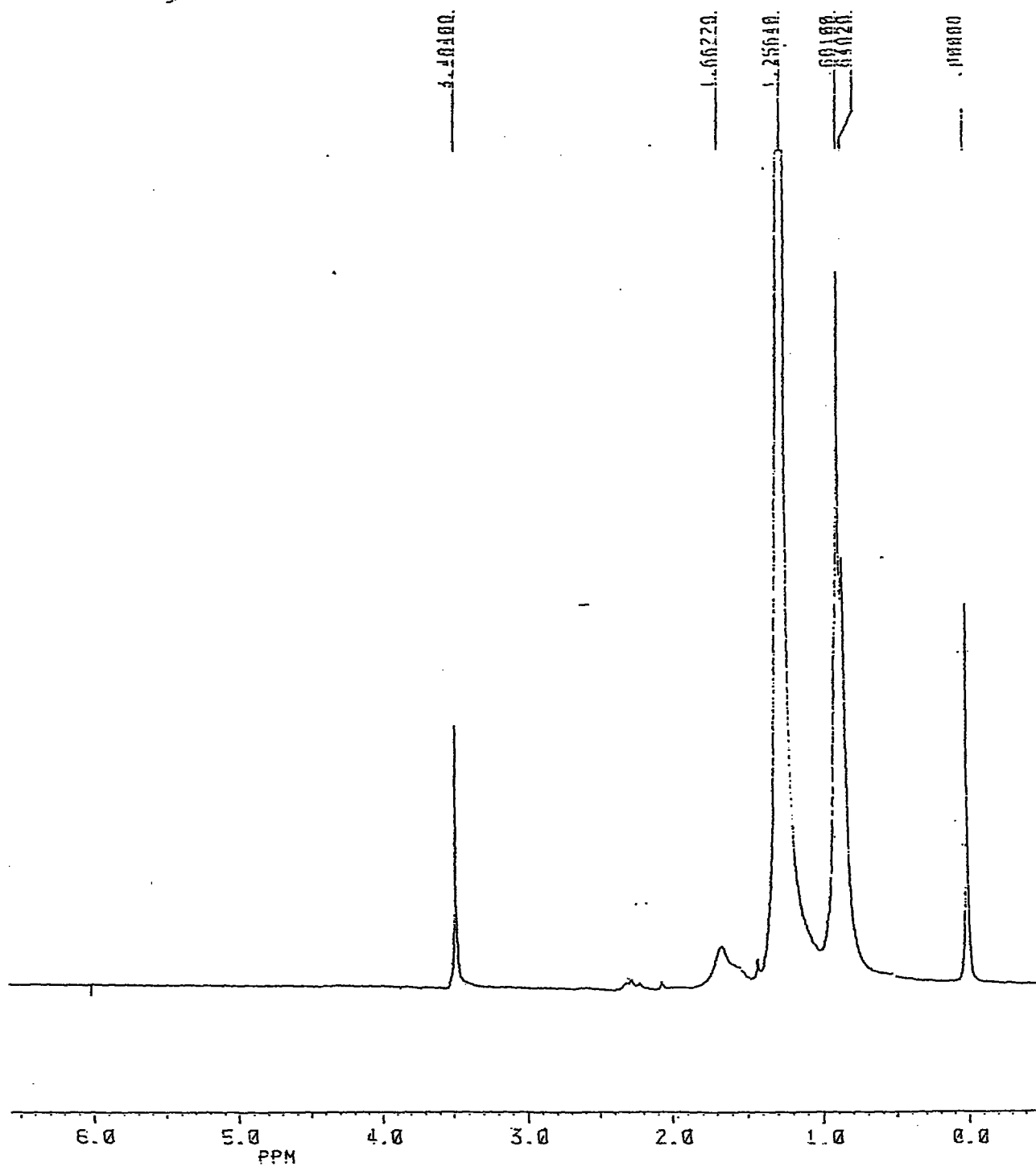


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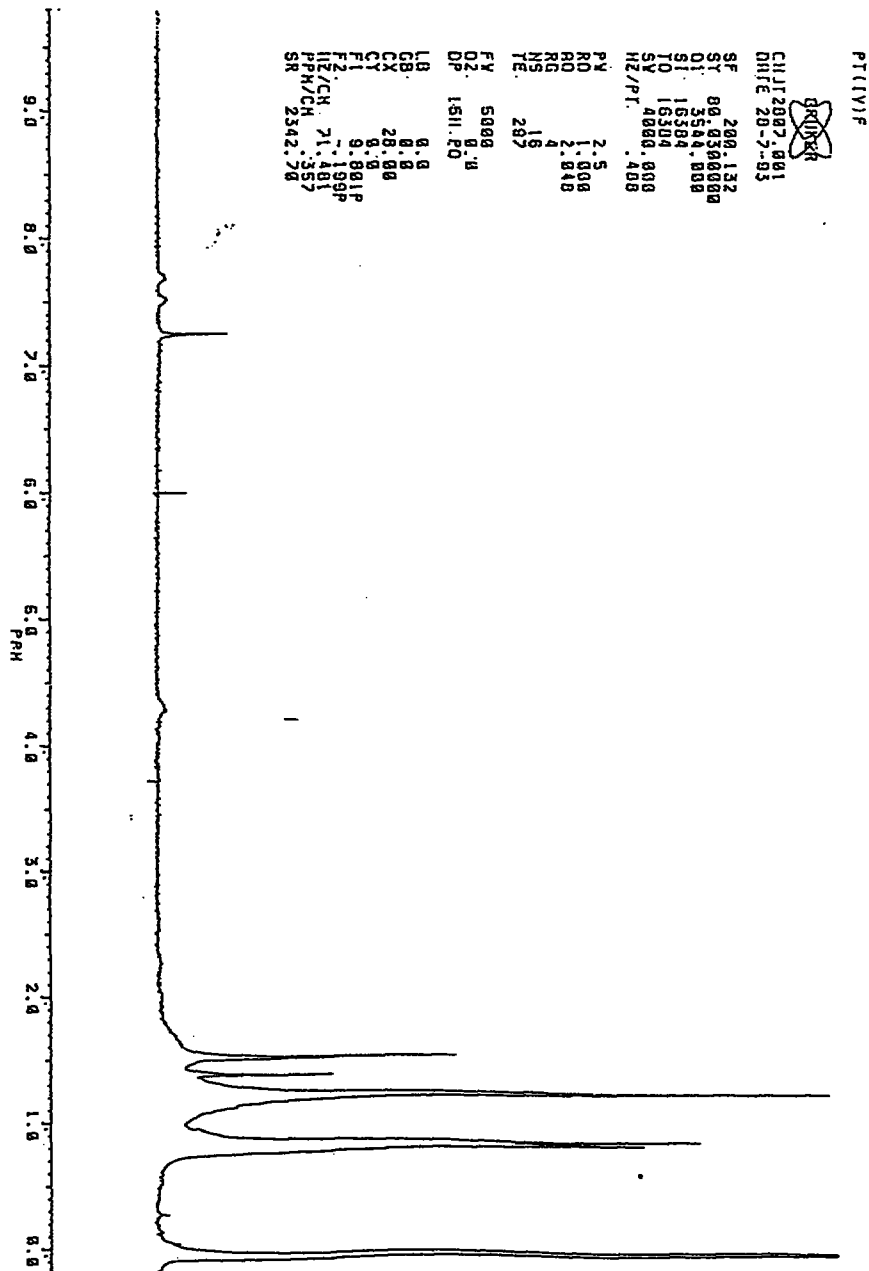
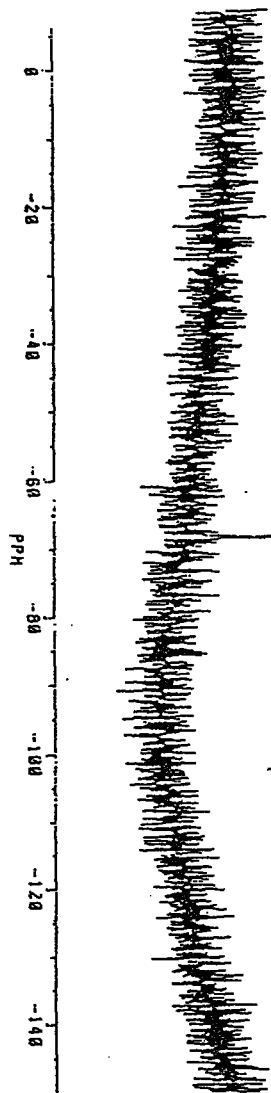


Fig 18

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Fig 19

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CHJ2909.009
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~~EXHIBIT~~

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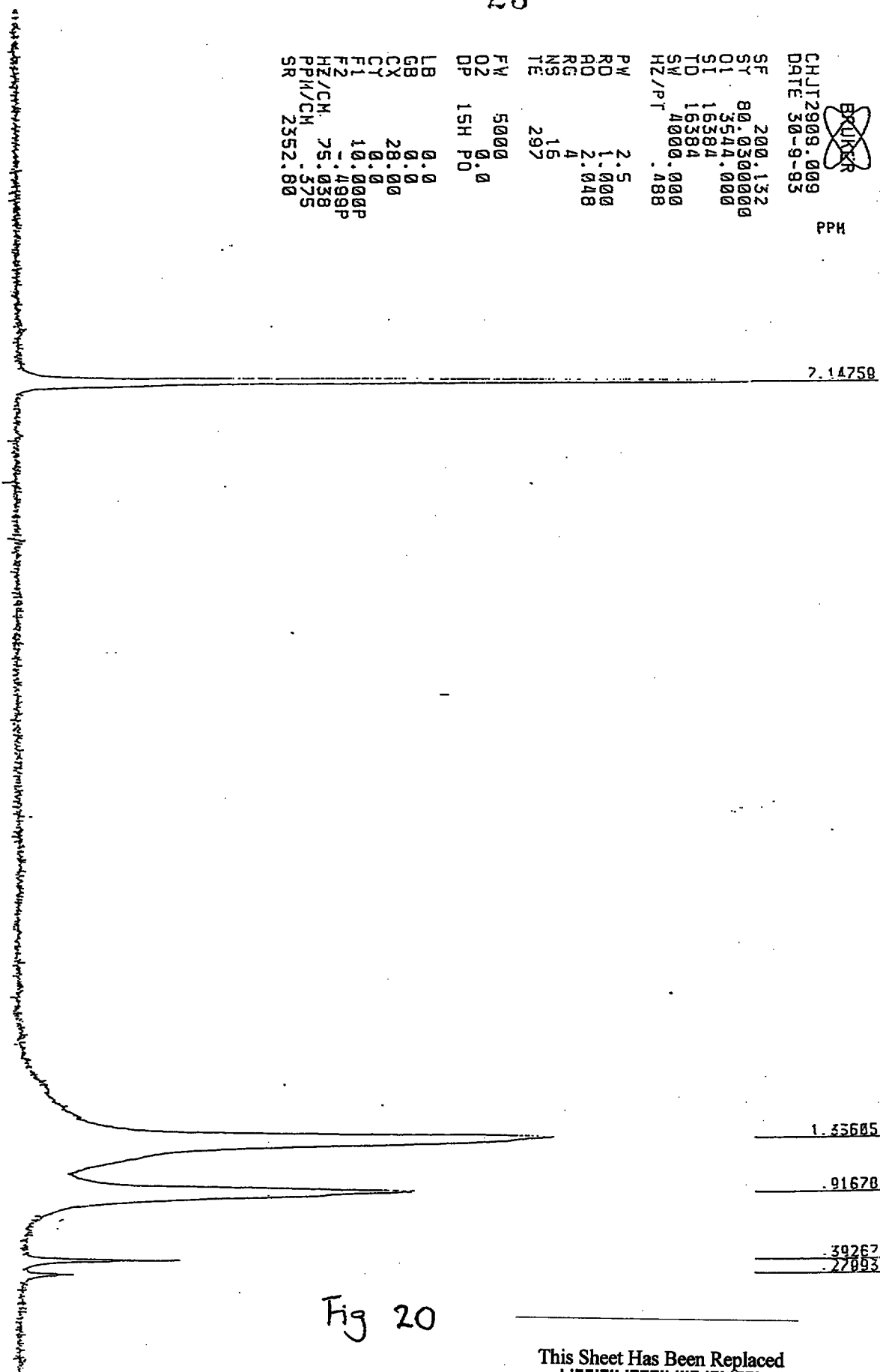


Fig 20

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SW	4000.000
HZ/PT	.488
PV	2.5
RO	1.000
RG	2.048
NS	1
TE	16
	297
FV	5000
O2	0.0
DP	15H PO
LB	0.0
GB	0.0
CX	28.00
CI	14.00
F1	10.002P
F2	7.496P
HZ/CM	75.038
PPM/CM	.575
SR	2352.31

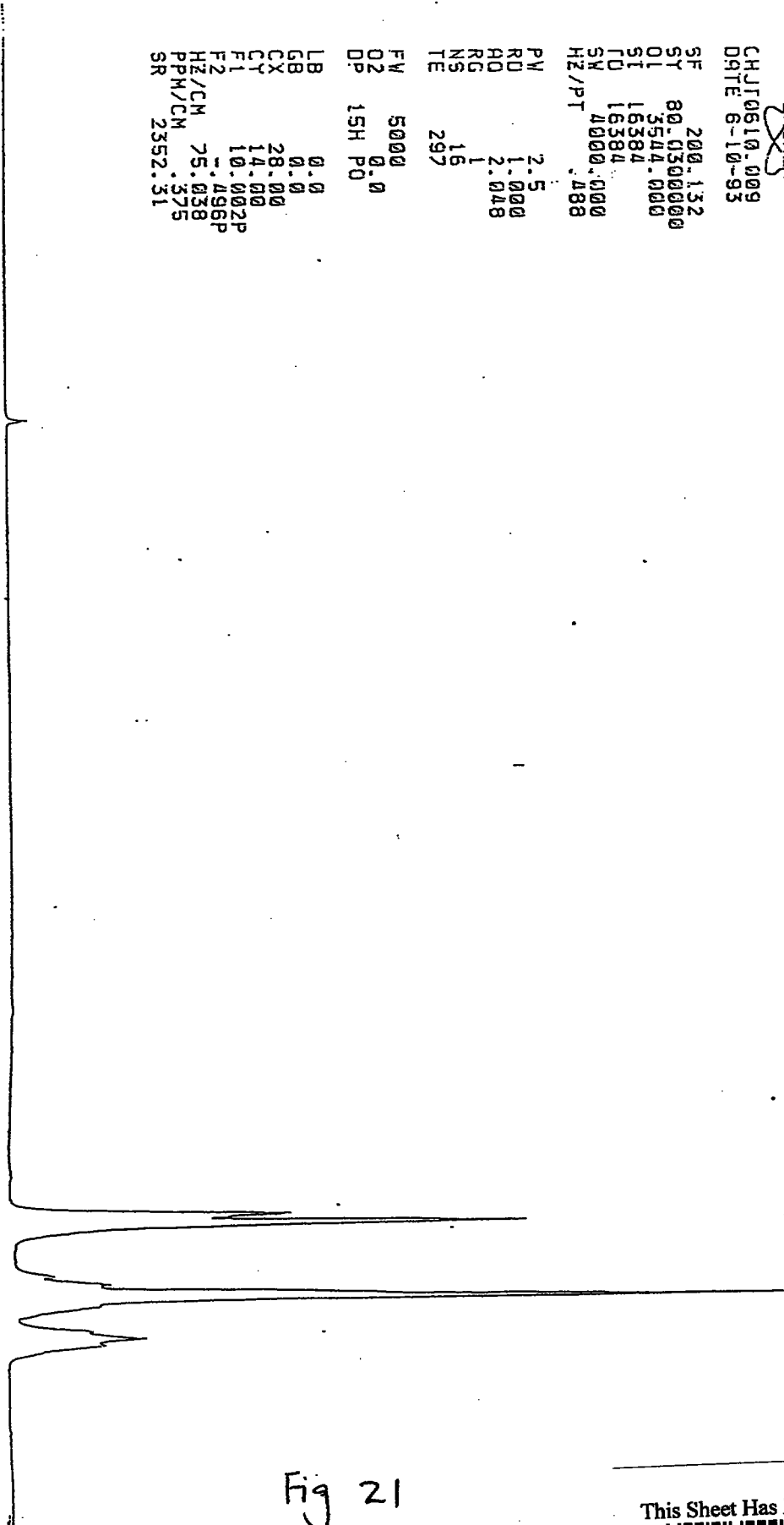


Fig 21

This Sheet Has Been Replaced



25

CHIT0510.016
 RU PROG:
 POKGATE. AUR
 DATE 7-10-93
 PPH

BRUKER

ST	50.323
SI	50.0300000
SI	6346.000
TD	16384
SV	15151.515
HZ/PT	1.650
PV	3.0
RD	0.0
RG	.541
NS	400
TE	401
	297
FW	19000
O2	3350.000
DP	20H CPD
LB	5.000
GB	0.0
CX	28.00
CT	14.00
F1	200.000P
F2	-10.009P
HZ/CM	377.440
PPH/CM	7.500
SR	3304.73

128.320
 122.639
 127.358

METHYL-CYCLOHEXYL PLATINUM FLUORIDE

38.368
 35.005
 33.784

22.181
 26.788

Fig 22

This Sheet Has Been Replaced



JHT0610.001
 DATE 6-10-93
 BXKER
 3E 200.132
 3Y 80.0300000
 3I 3544.000
 3L 16384
 3M 16384
 3W 4000.000
 3Z/PT .488
 3Y 2.5
 3D 1.000
 3C 2.048
 3S 1
 3E 16
 3V 297
 3W 5000
 3Z 0.0
 3P 15H PO
 3B 0.0
 3X 0.0
 3Y 28.00
 3I 0.0
 3L 10.000P
 3M 1.499P
 3W 75.038
 3Z/CM .375
 3P/CM .375
 3R 2352.31

PPH

8.83788

7.15205

6.58119
 6.58513
 6.28627
 6.28808
 5.98362
 5.96666
 5.96172
 5.95442
 5.94714
 5.65866
 5.61263
 5.57700

2.20505

1.28163
 1.23166

.98135
 .67201
 .64060
 .55069
 .43840
 .38471

Fig 23

This Sheet Has Been Replaced



GRUBER
 CHJ0510.014
 AU PROG:
 POCGATE.9UR
 DATE 7-10-93

PPM

SF 50.323
 ST 50.030000
 01 8346.000
 ST 16384
 TD 16384
 SW 15151.515
 HZ/PT 1.650

PW 3.0
 RD 0.0
 RG 0.541
 NS 400
 TE 297

FV 19000
 OZ 3350.000
 DP 20H CPD

LB 5.000
 GB 0.0
 CX 28.00
 CY 14.00
 F1 200.000P
 F2 10.000P
 HZ/CM 377.440
 PPM/CM 7.500
 SR 3304.75

128.3
 127.6
 127.2

34.3

22.5

14.0

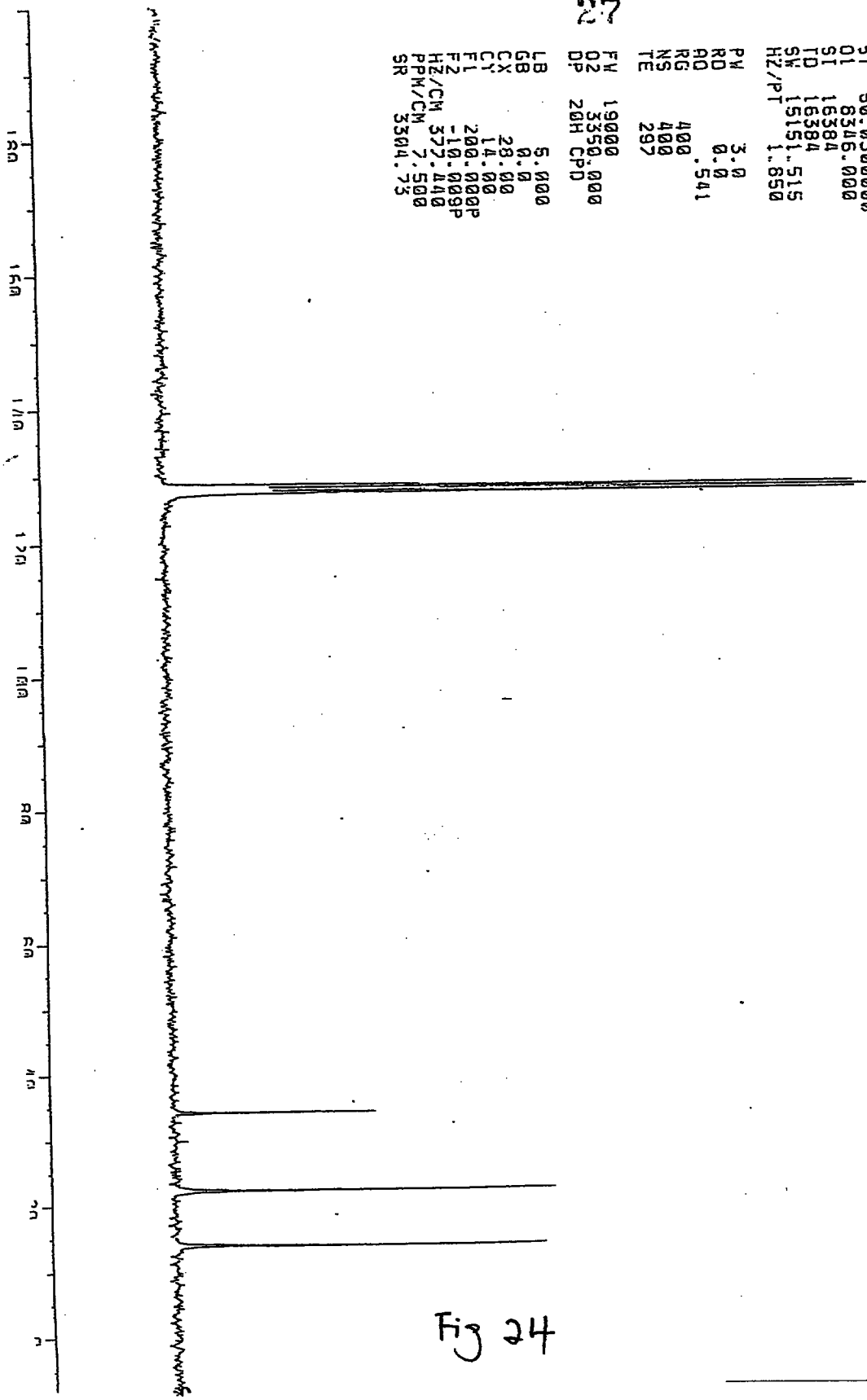


Fig 24

This Sheet Has Been Replaced



#1 10.006P
 #2 -150.000P
 #3 1.076E3
 #4 5.714
 #5 435.49

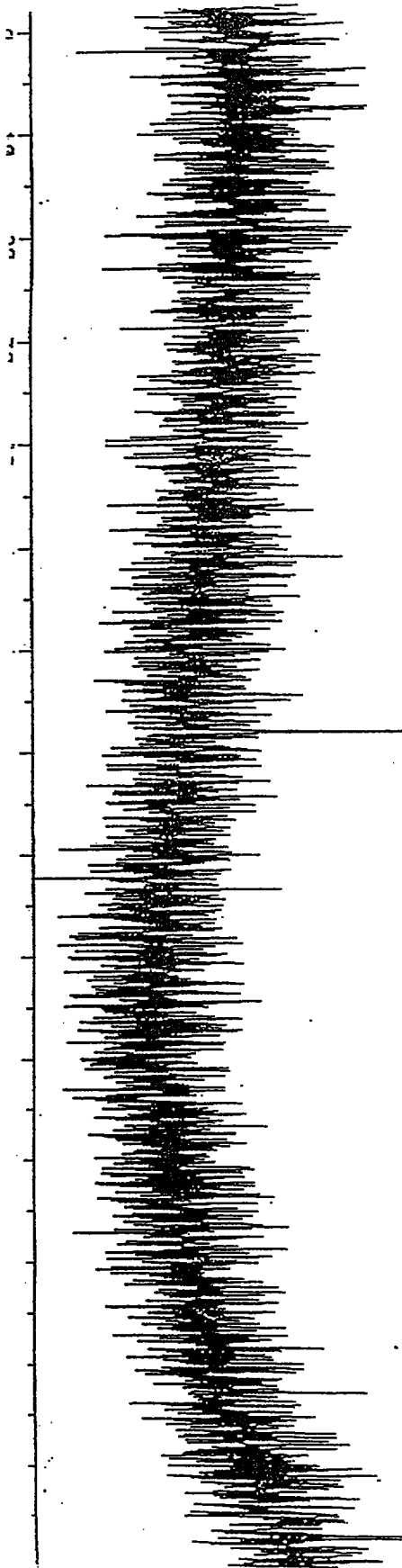


Fig 25

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1

REPLACEMENT SHEETS		SG
☐	Description	: paged to paged
☐	Claim(s)	: paged to paged
☒	Drawing(s)	: paged to paged 28.
☐	Abstract	: paged to paged
DATE OF RECEIPT		29/11/97

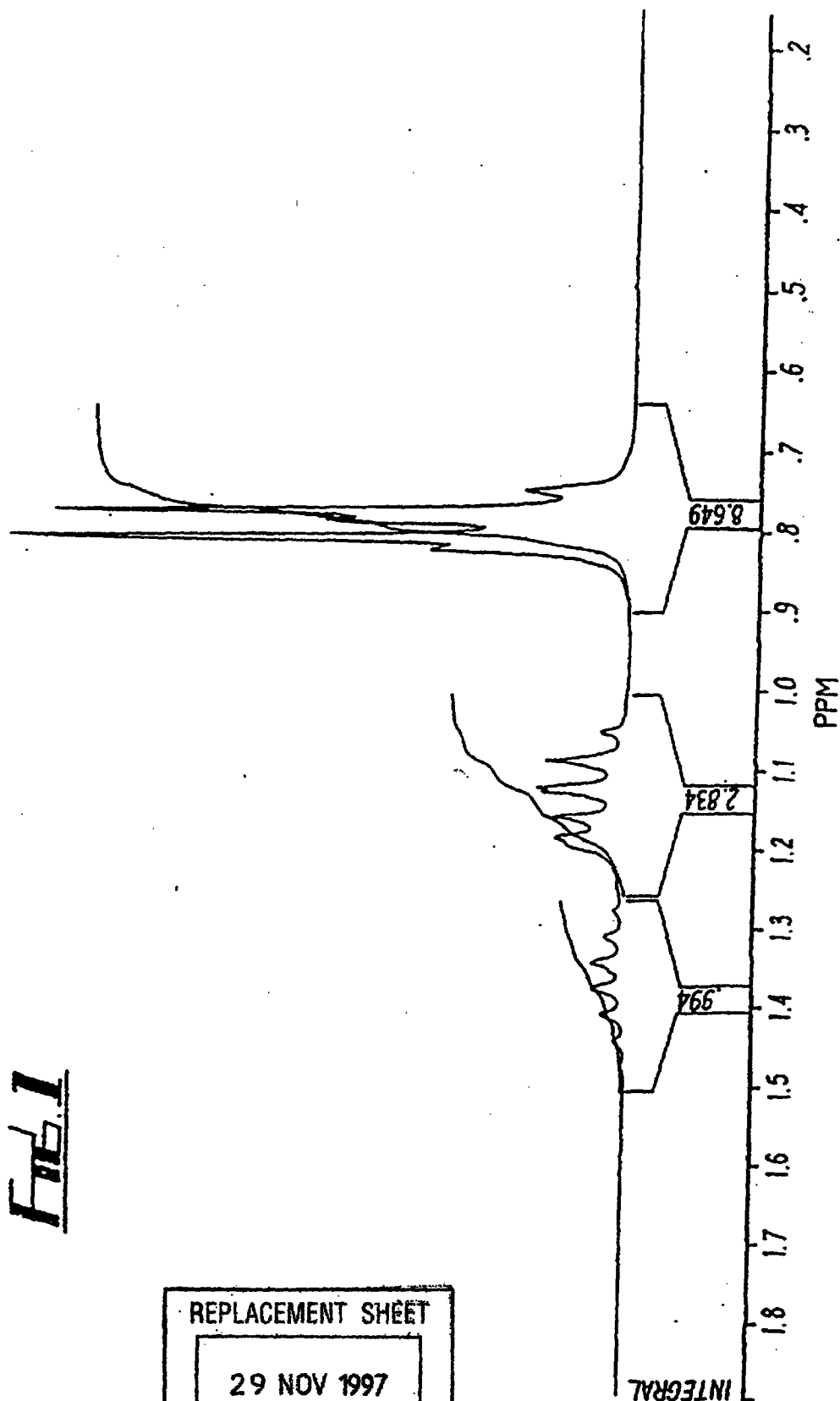


Fig. 1

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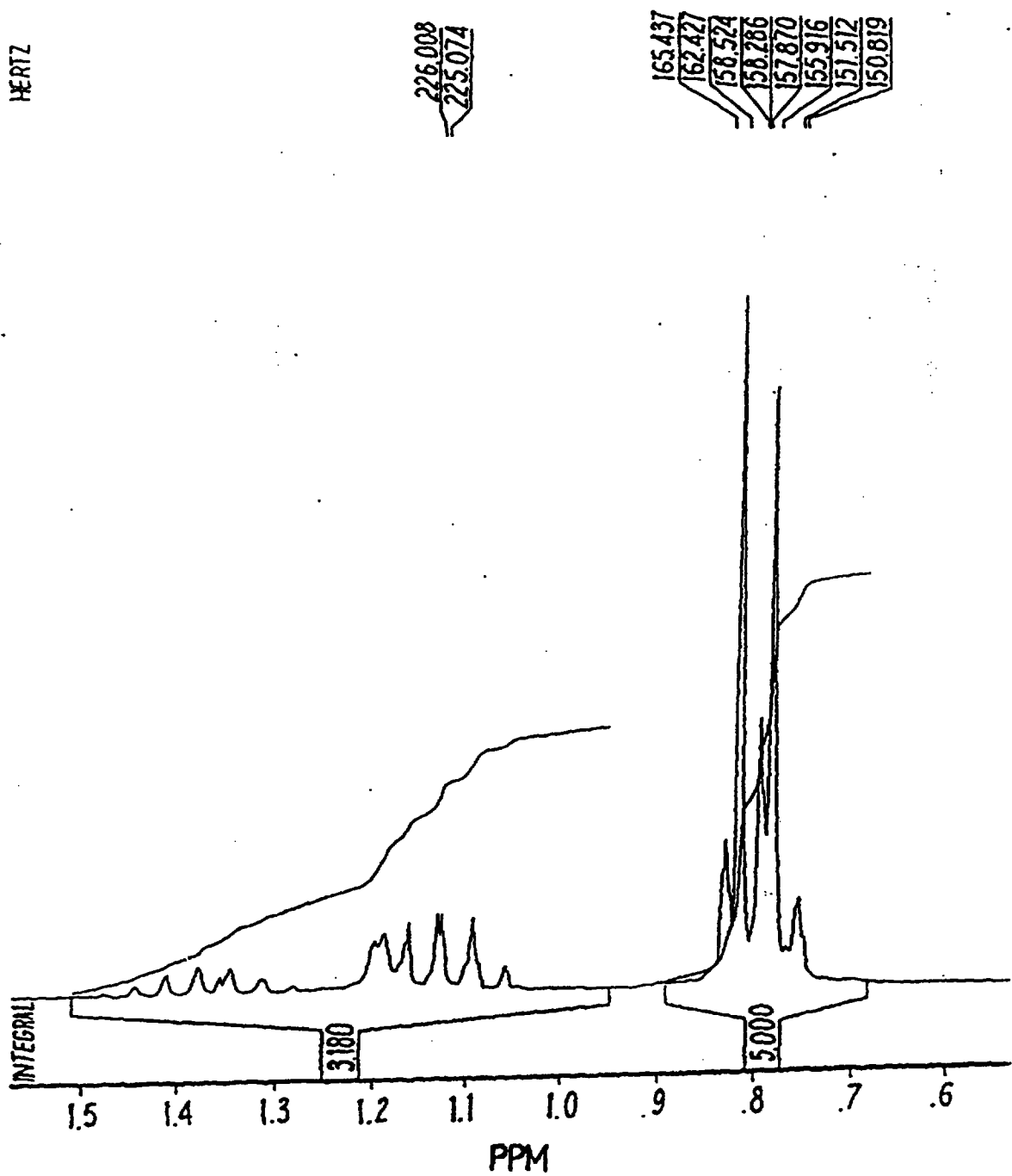


Fig. 1a

REPLACEMENT SHEET

29 NOV 1997

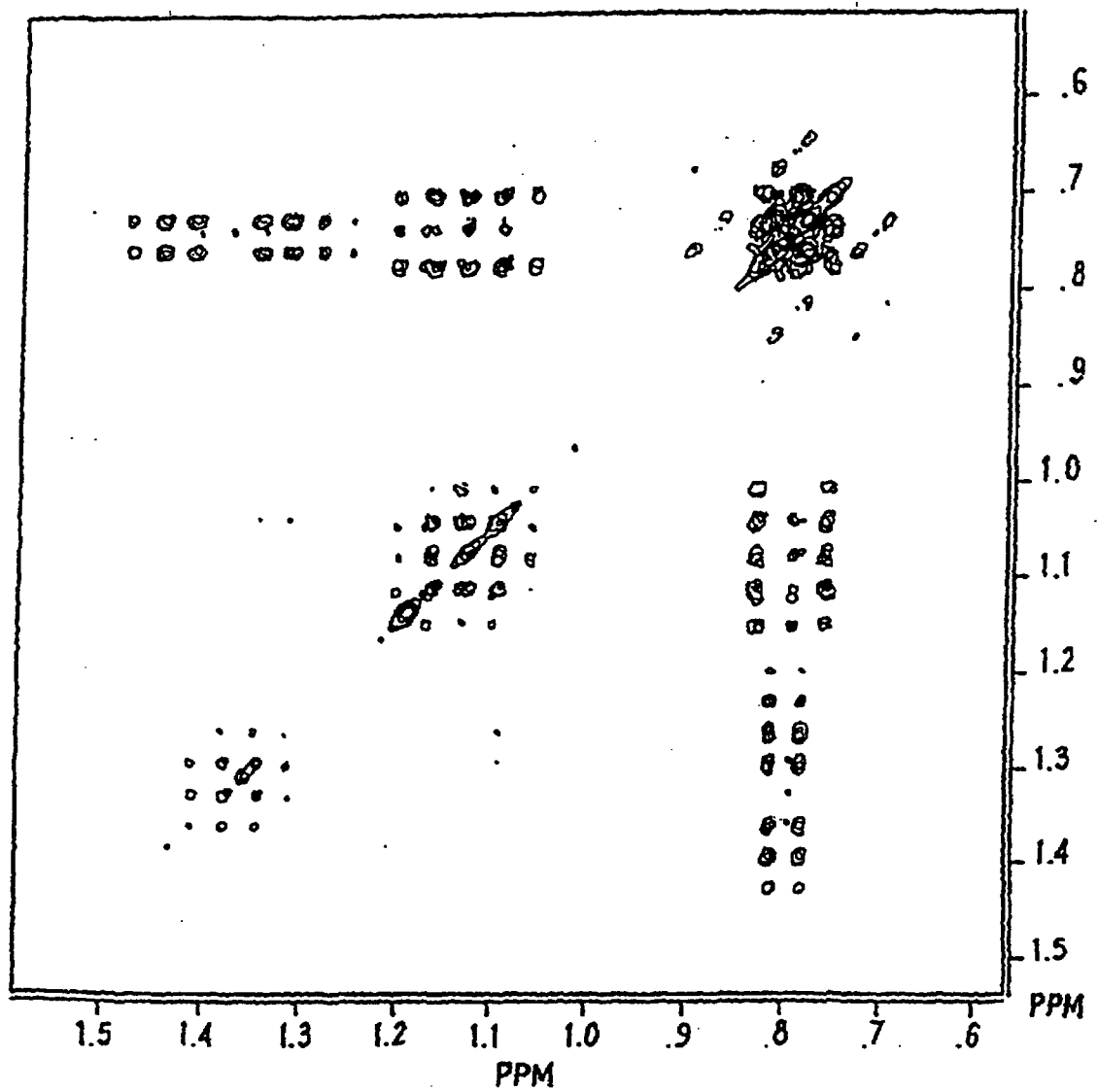
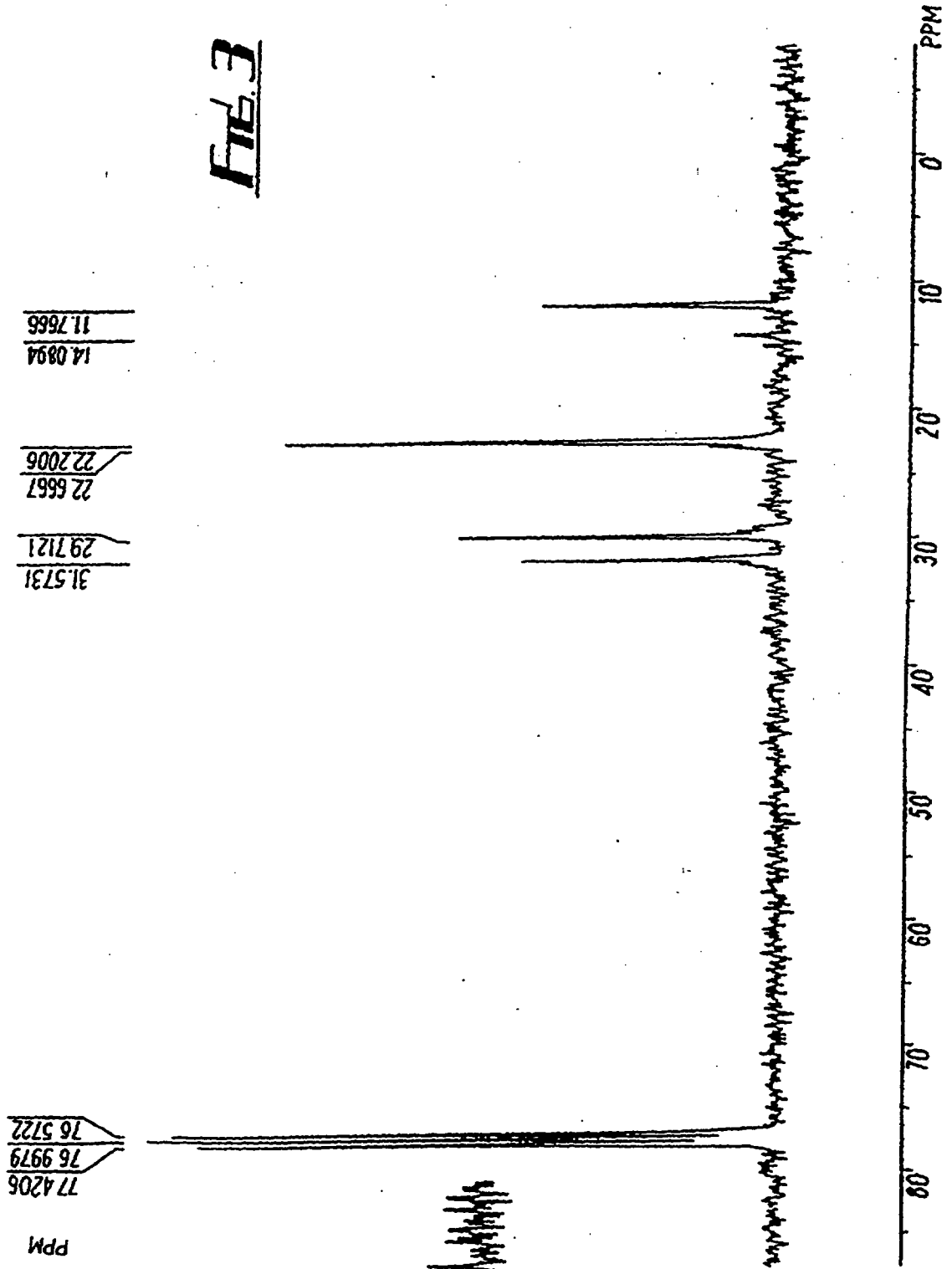


FIG. 2

REPLACEMENT SHEET

29 NOV 1997

Fig. 3



REPLACEMENT SHEET

29 NOV 1997

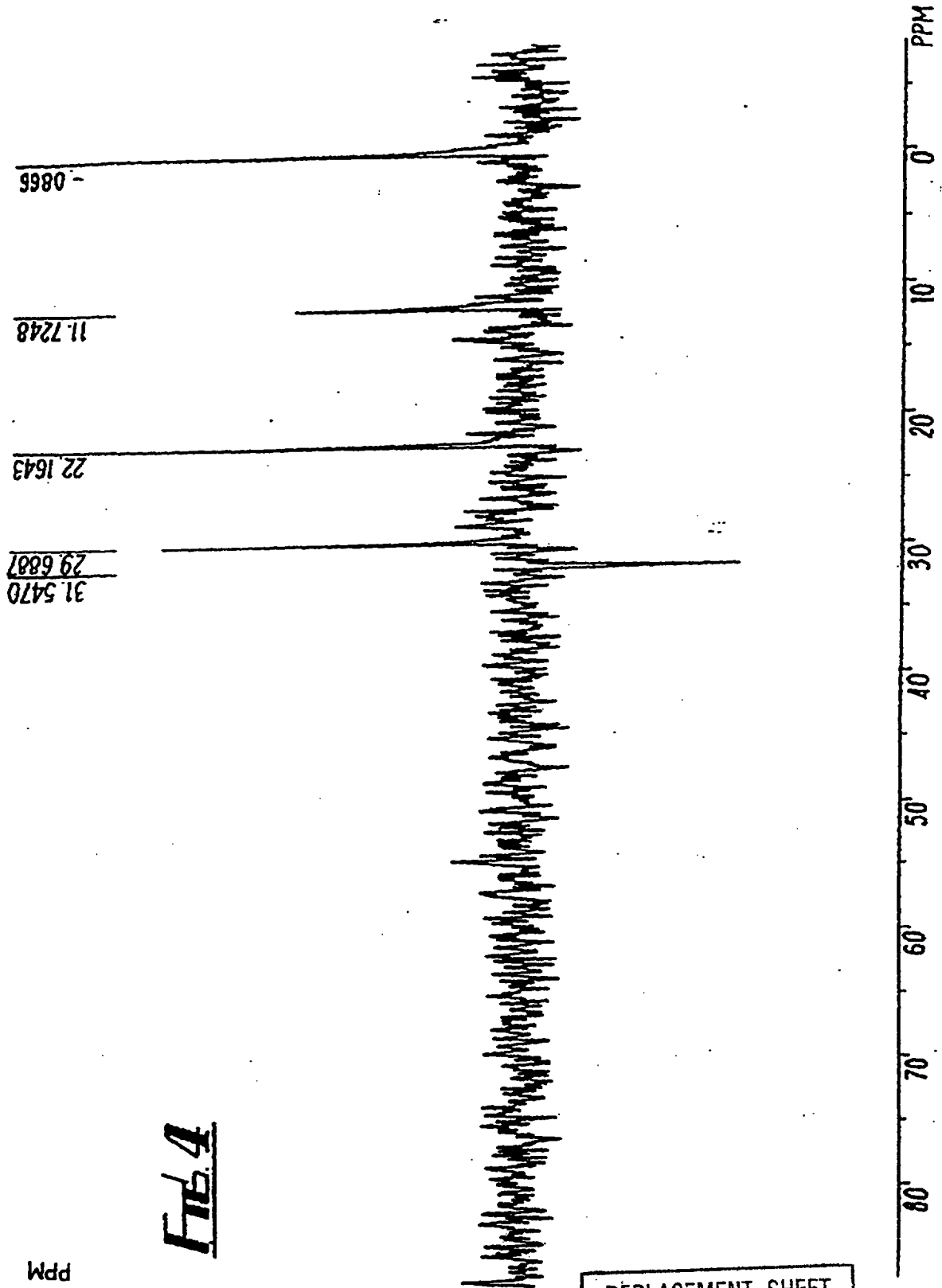


Fig. 4

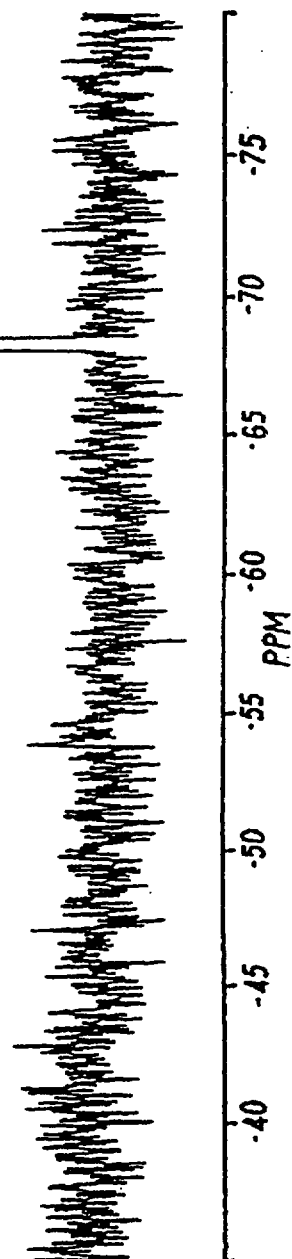
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File 5

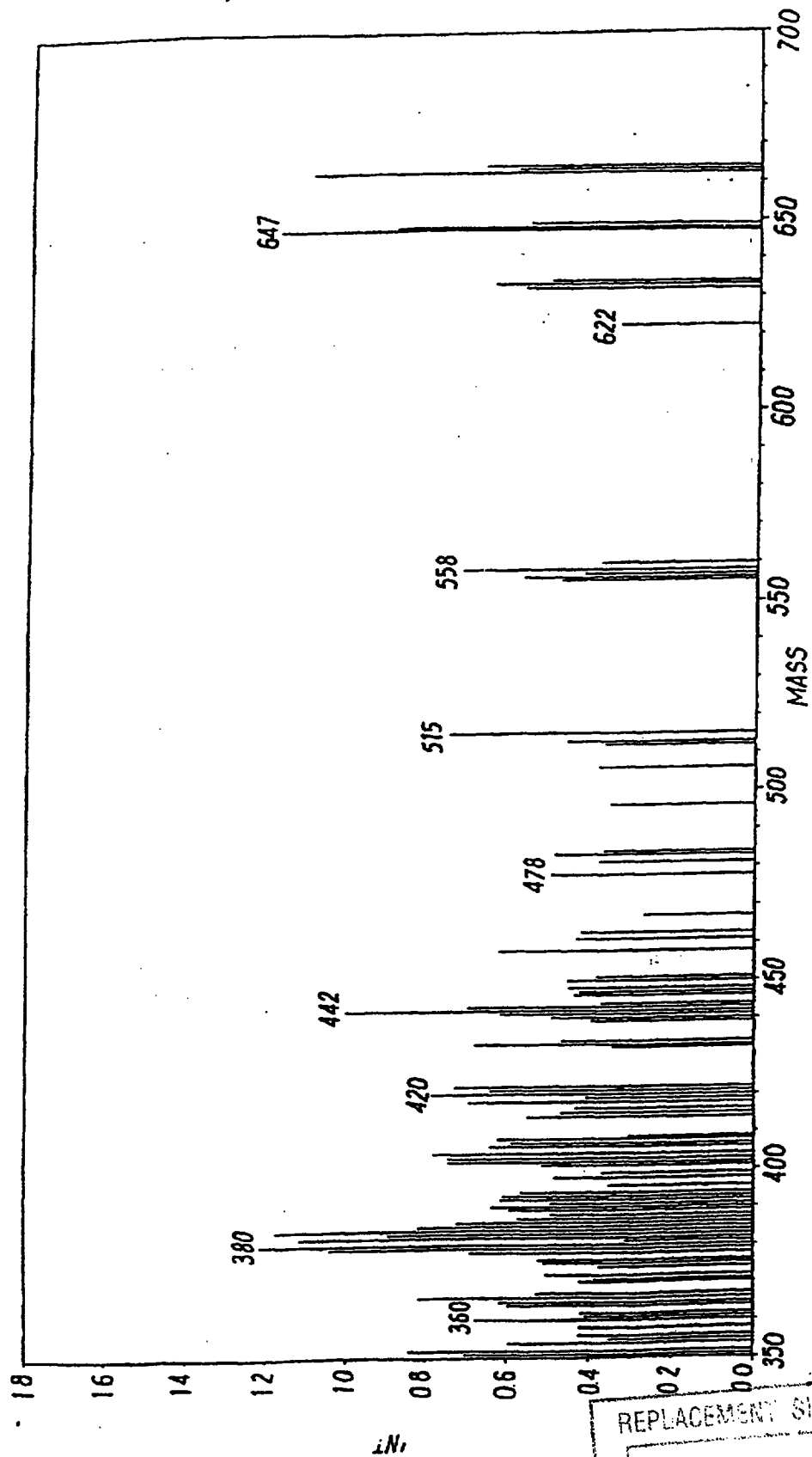
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ppm



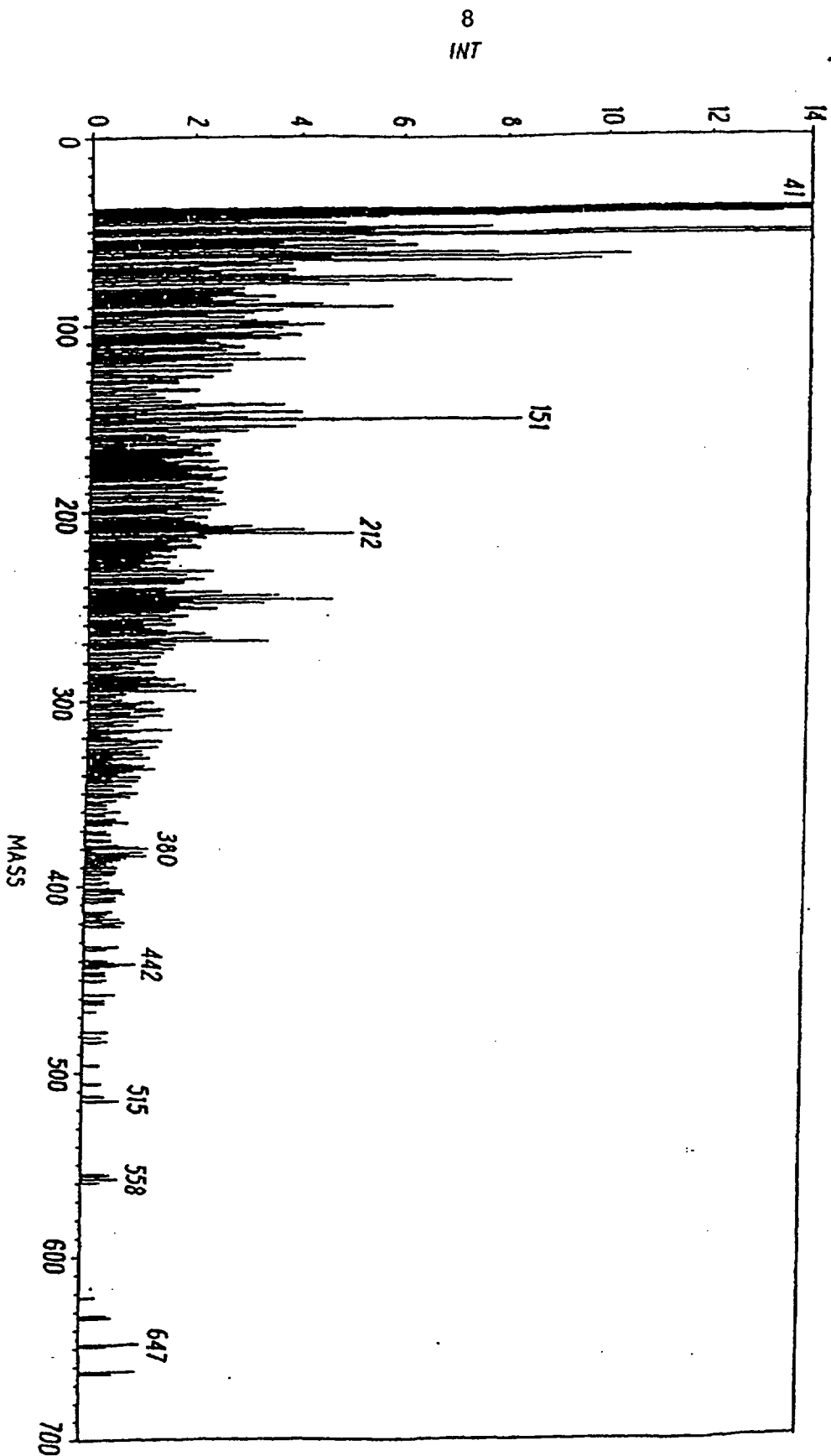
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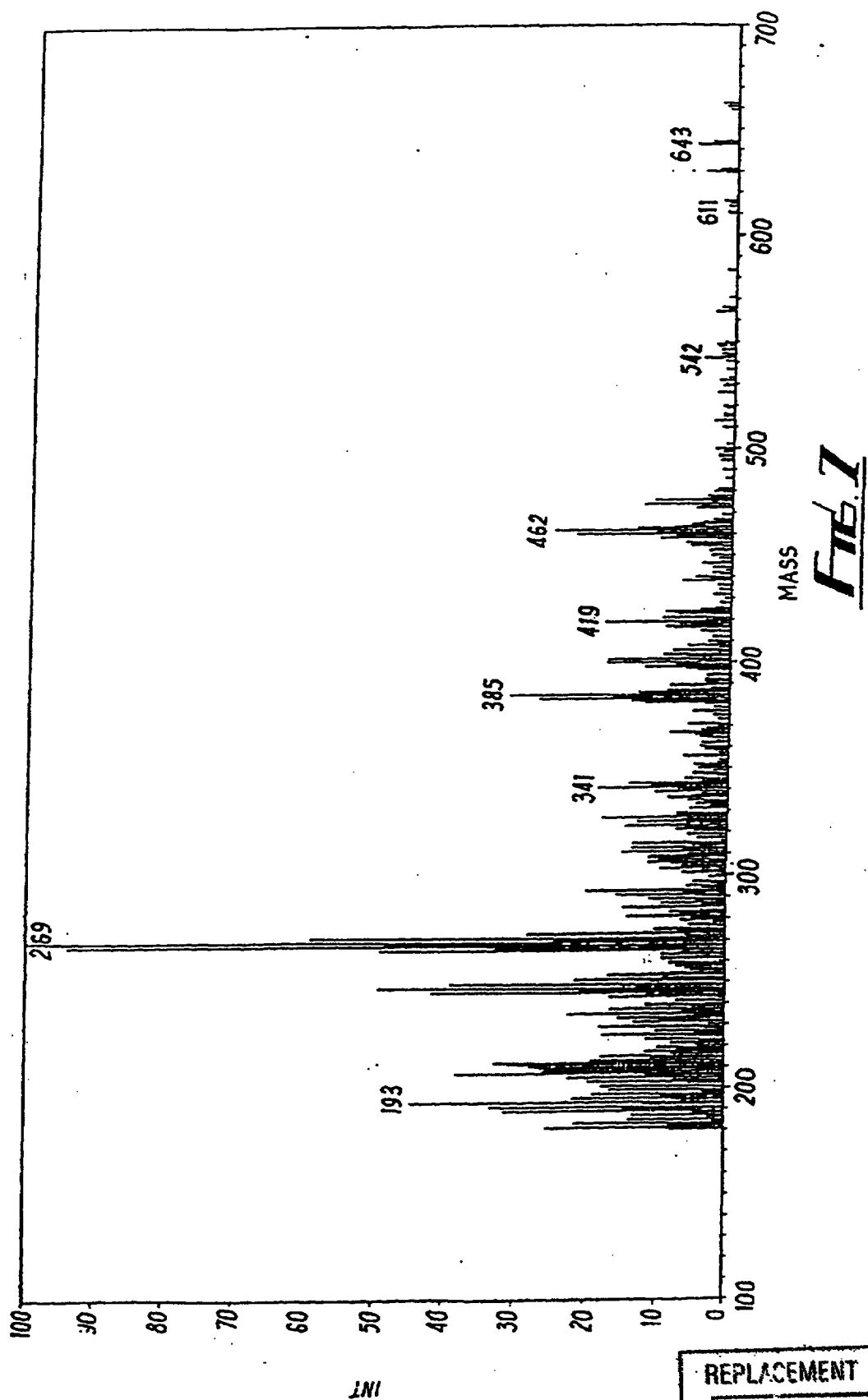
REPLACEMENT SHEET
29 NOV 62



End. 6a

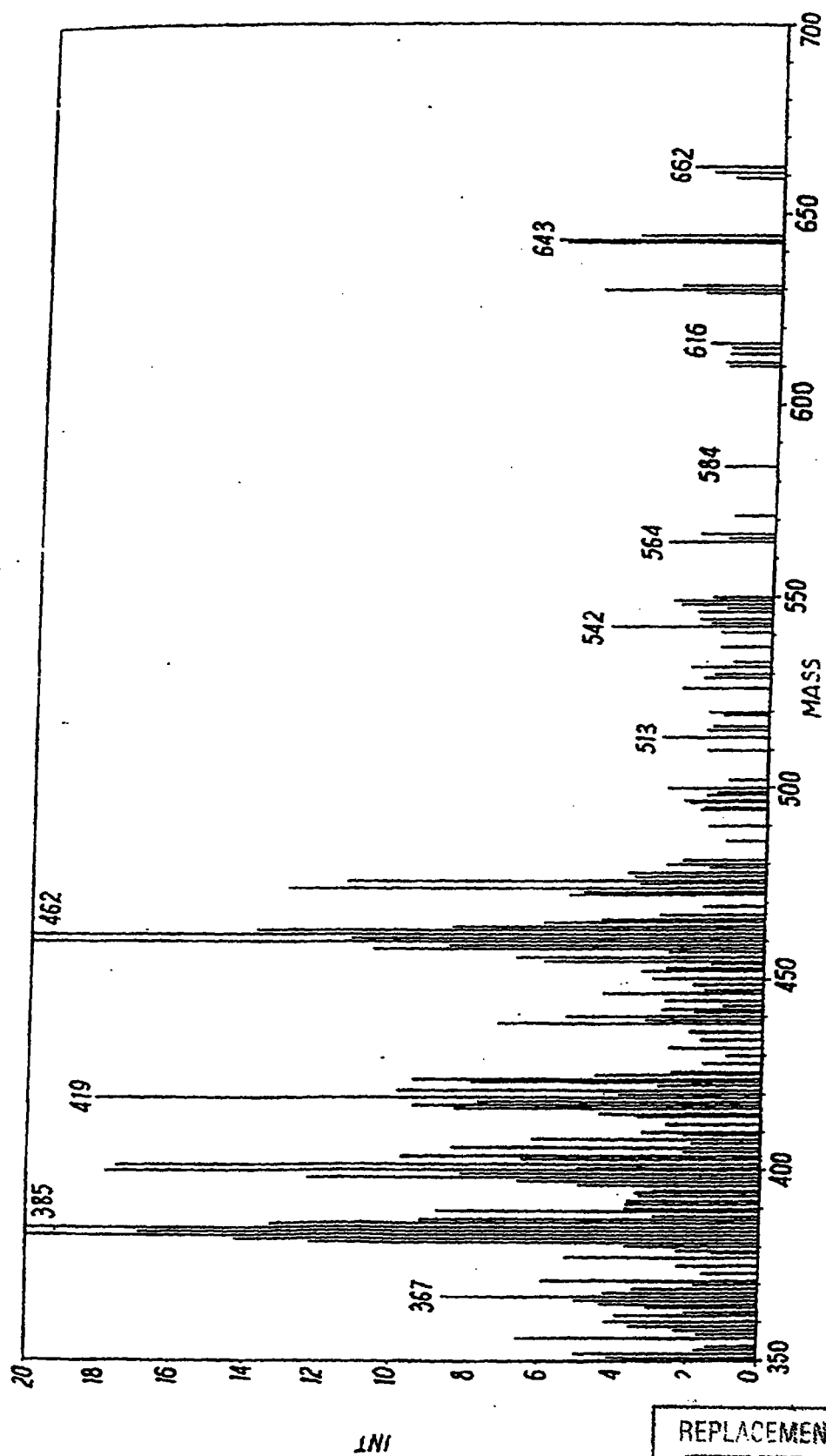
REPLACEMENT SHEET

29 NOV 1997



REPLACEMENT SHEET

29 NOV 1997

**Fig. 7a**

REPLACEMENT SHEET

29 NOV 1997

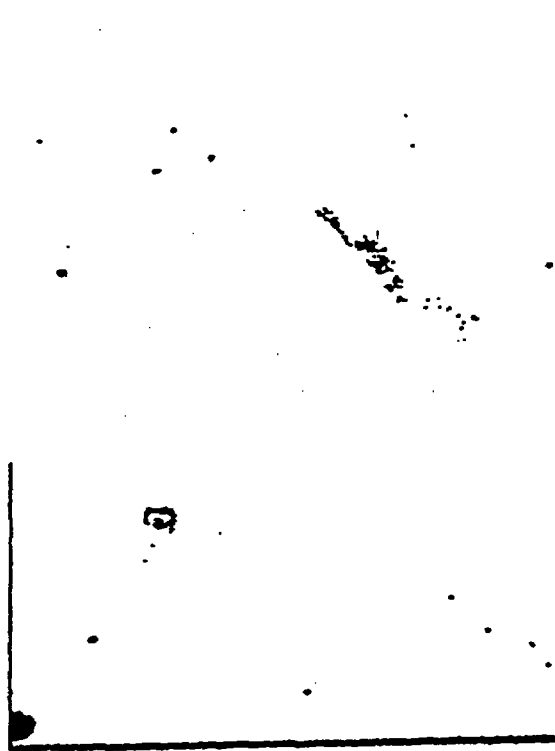
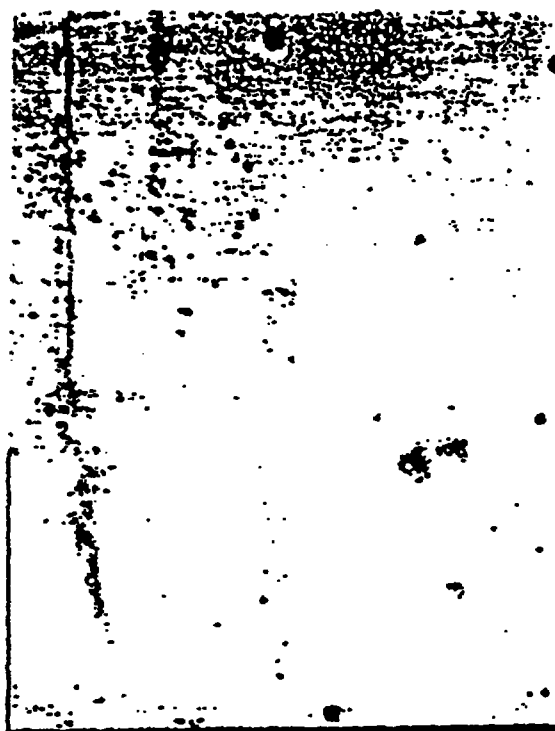
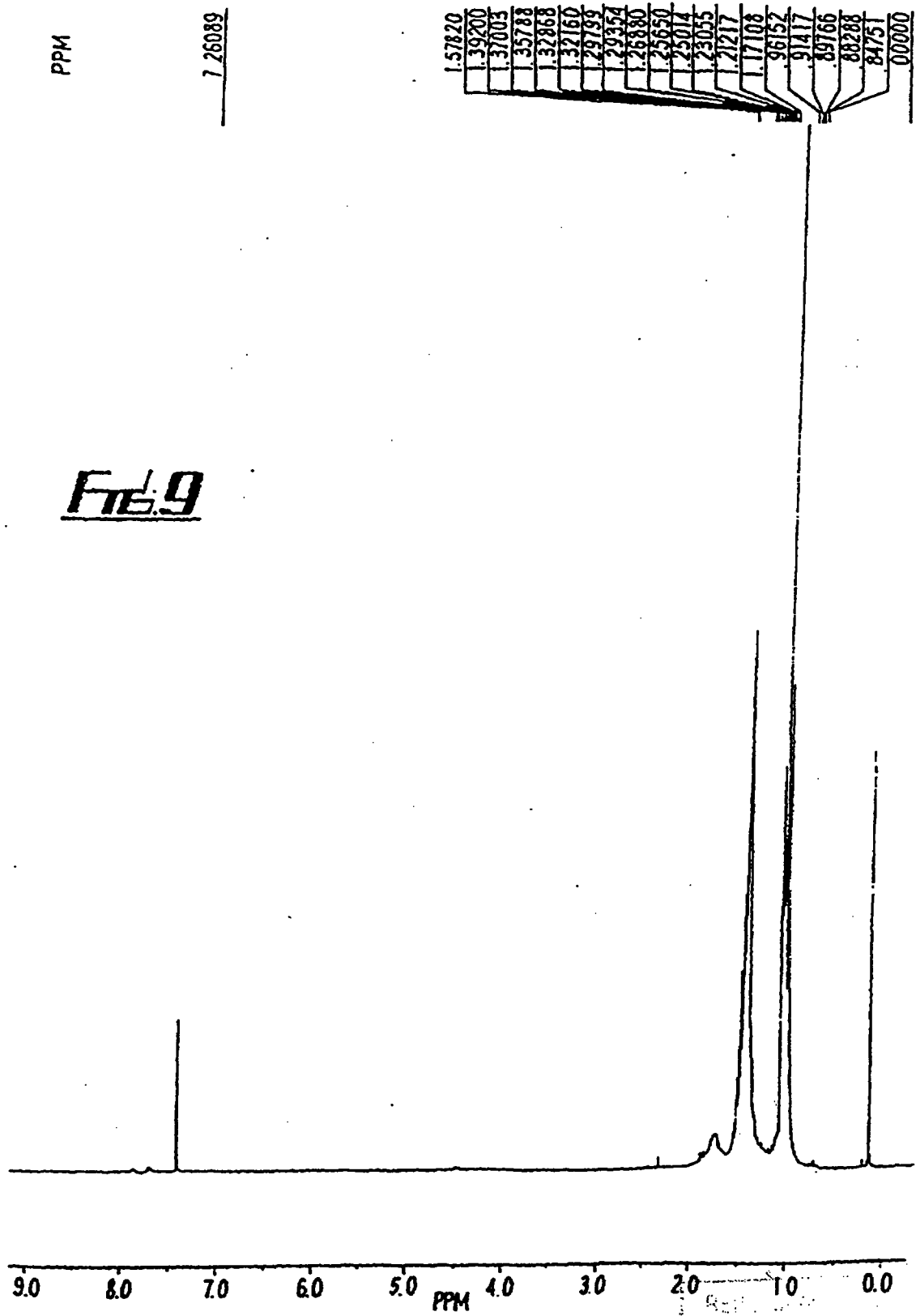


Fig. 8

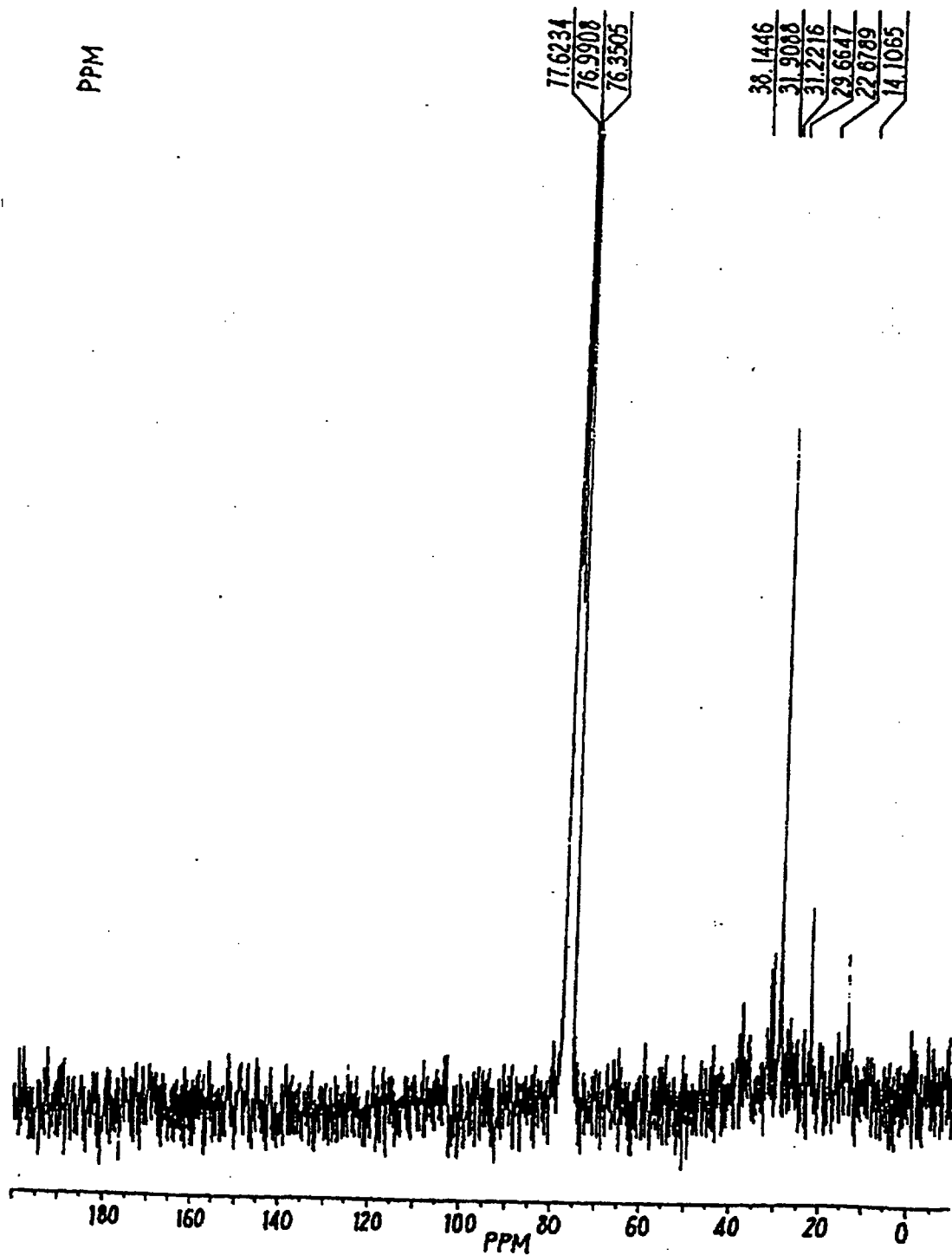


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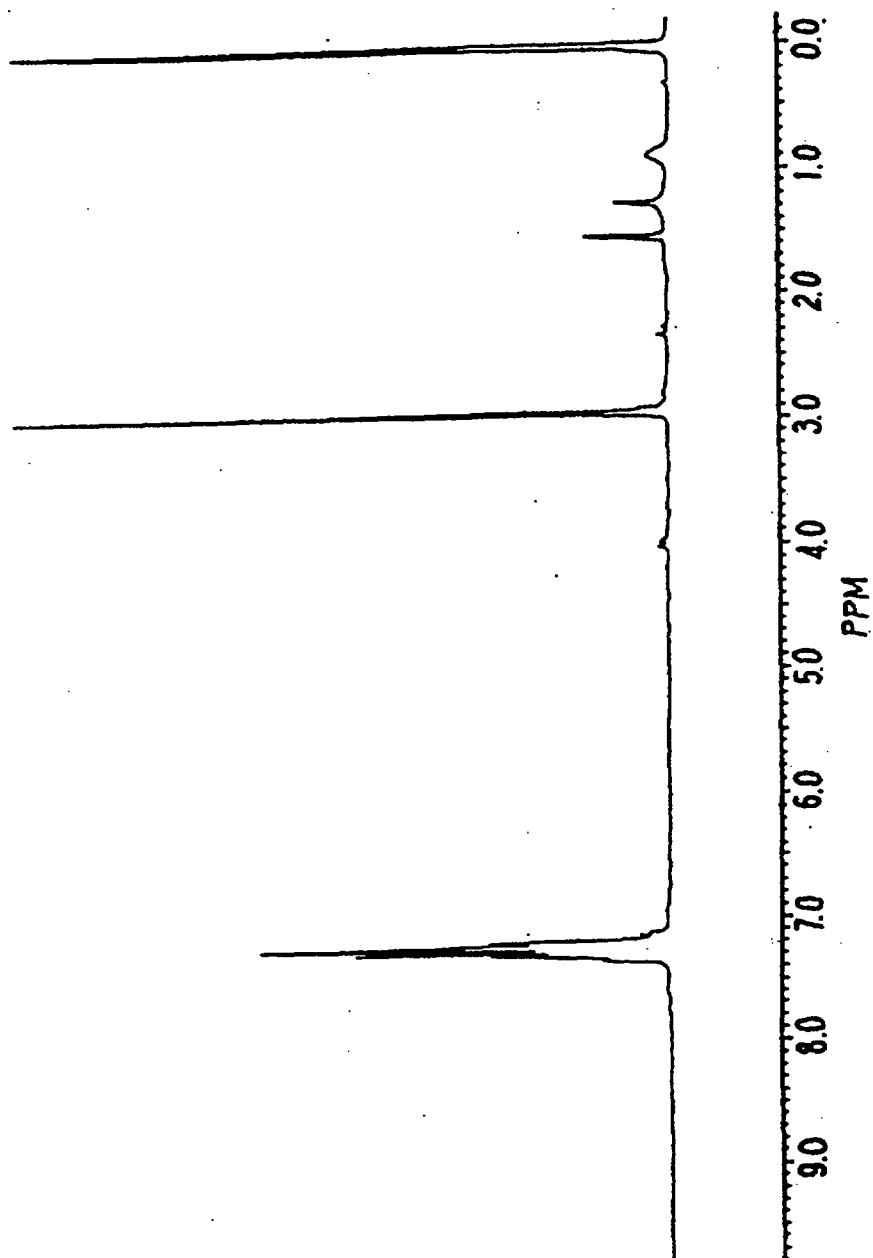


29 NOV 1997

Fig. 10

REPLACEMENT SHEET

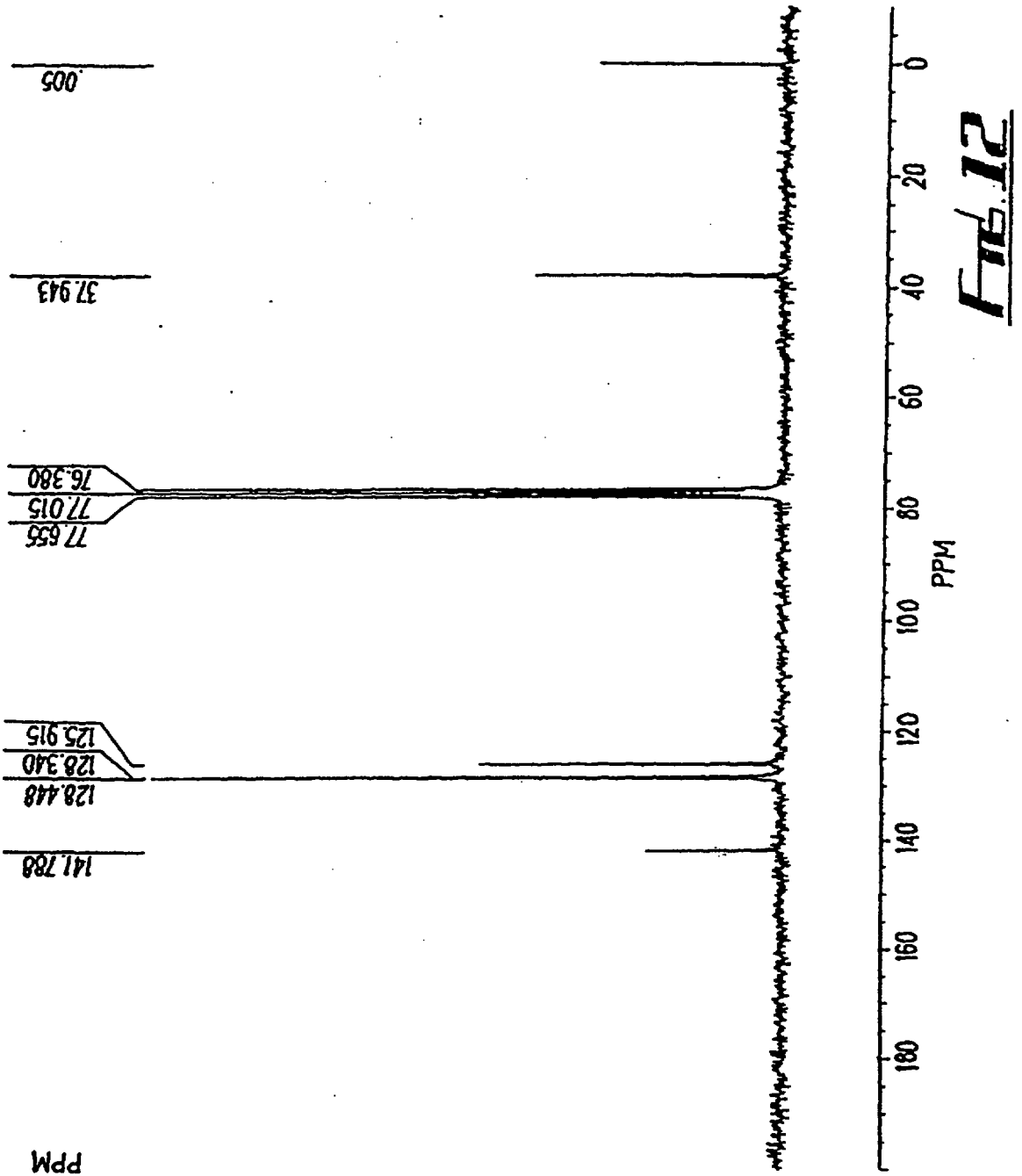
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File 11

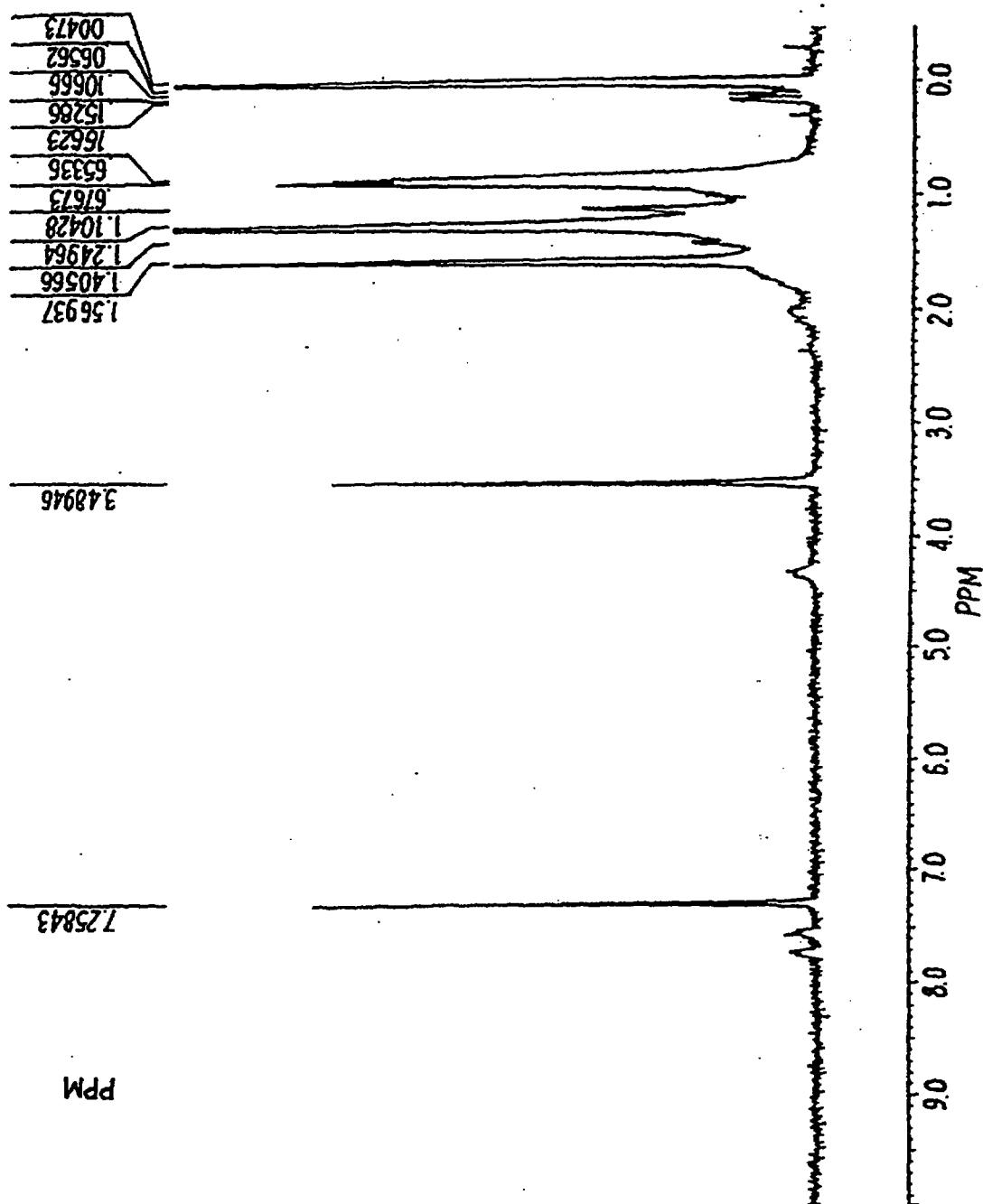
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REPLACEMENT SHEET

29 NOV 1997



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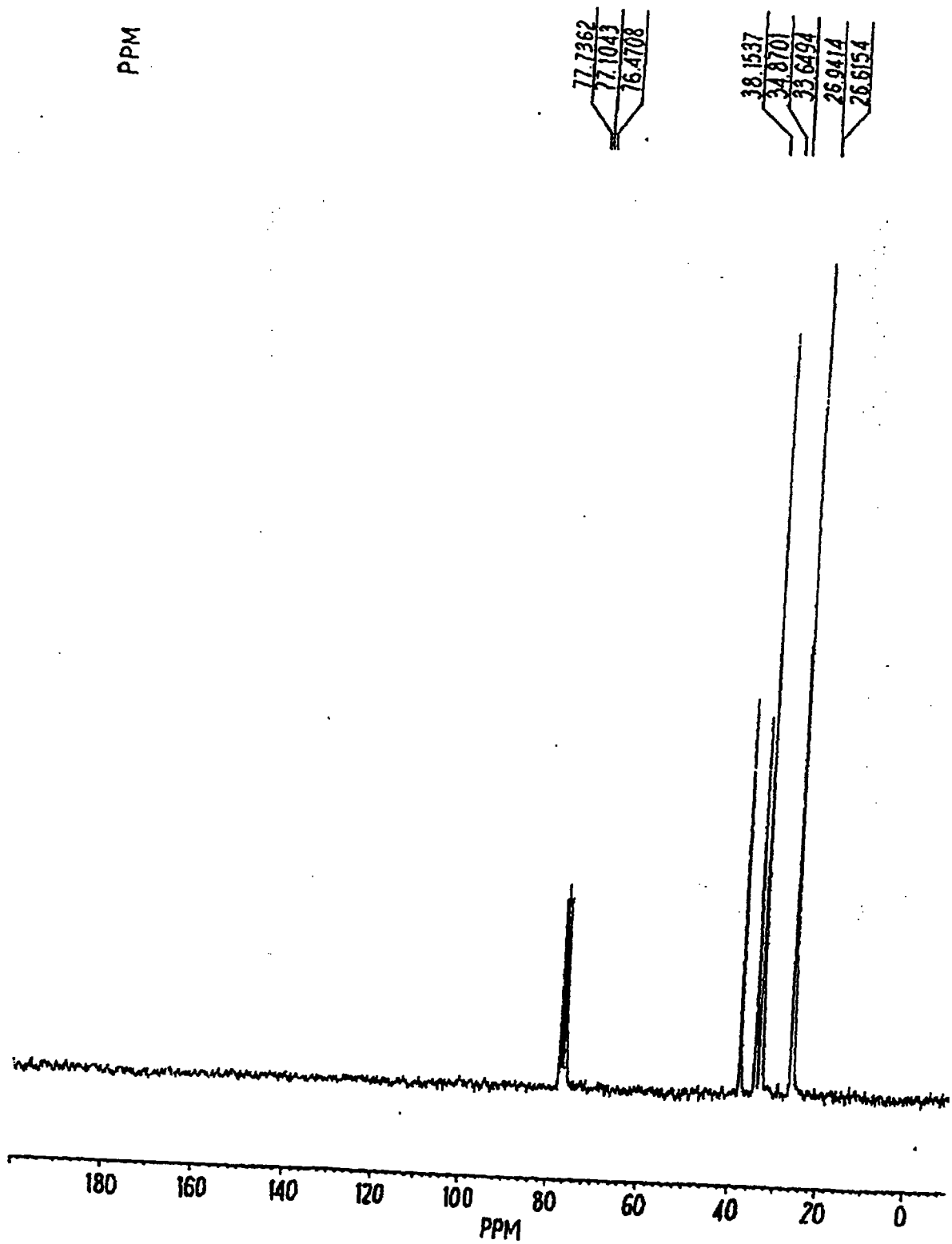


FIG. 14 REPLACEMENT SHEET

29 NOV 1997

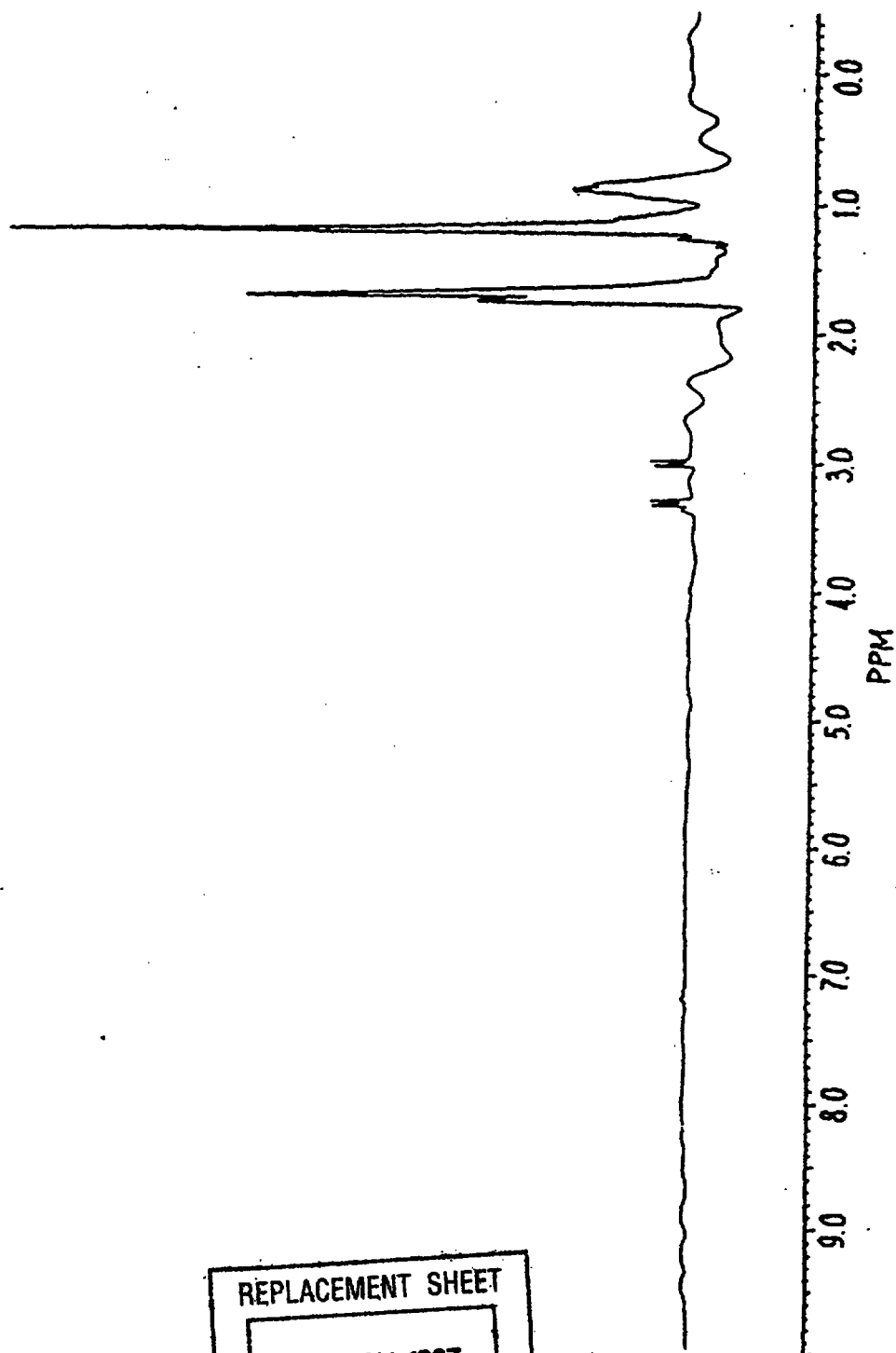


Fig 15

REPLACEMENT SHEET

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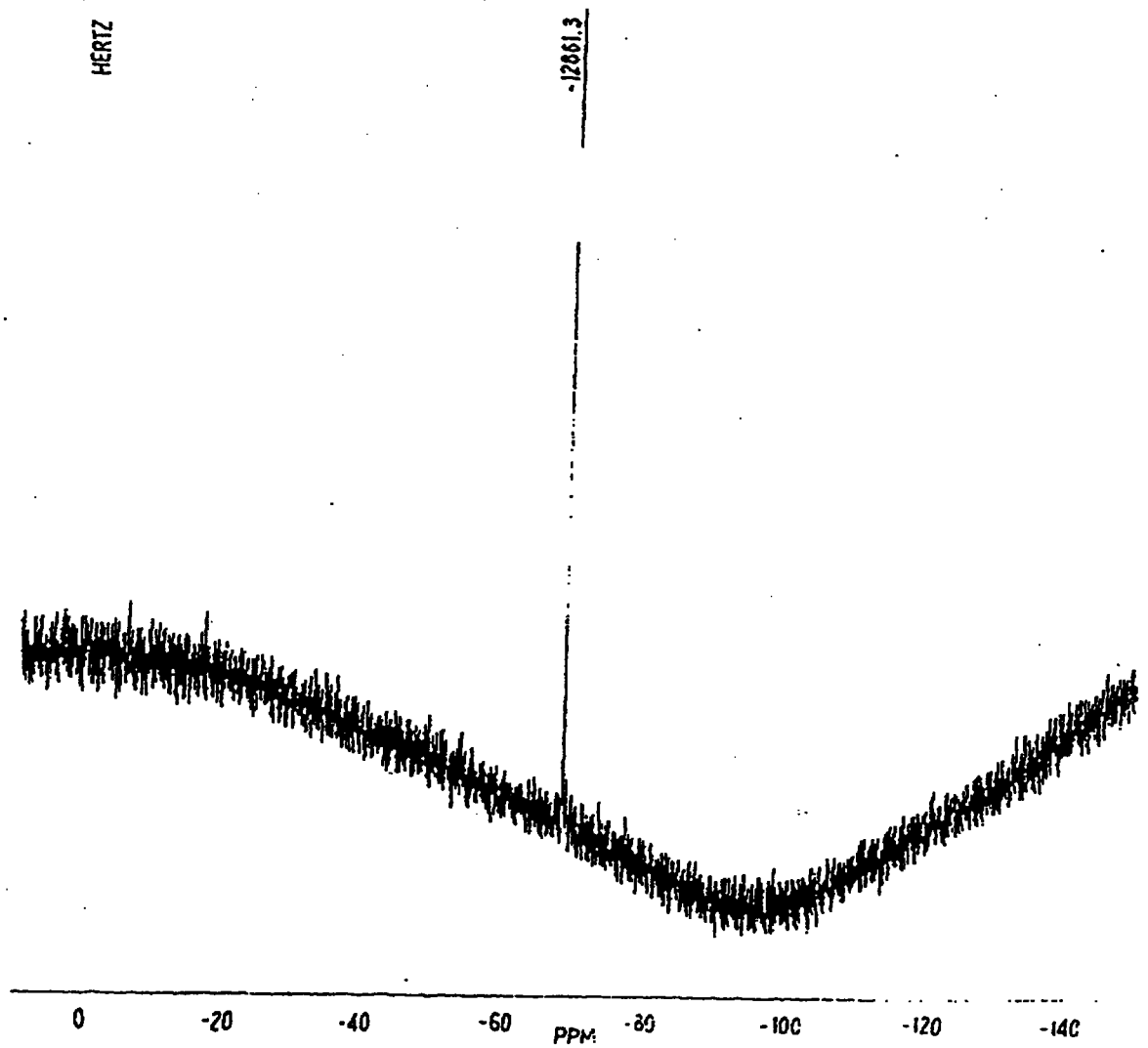
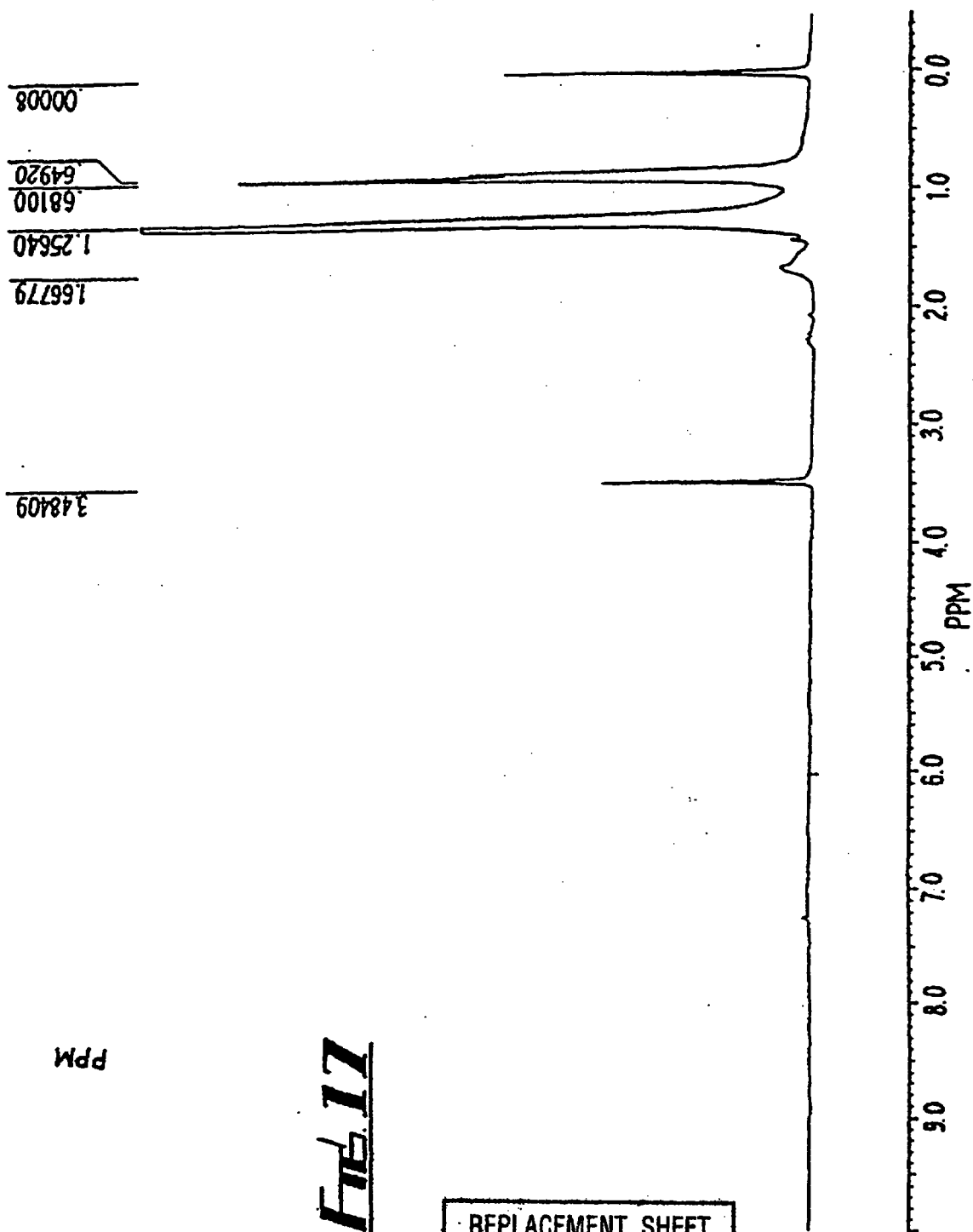
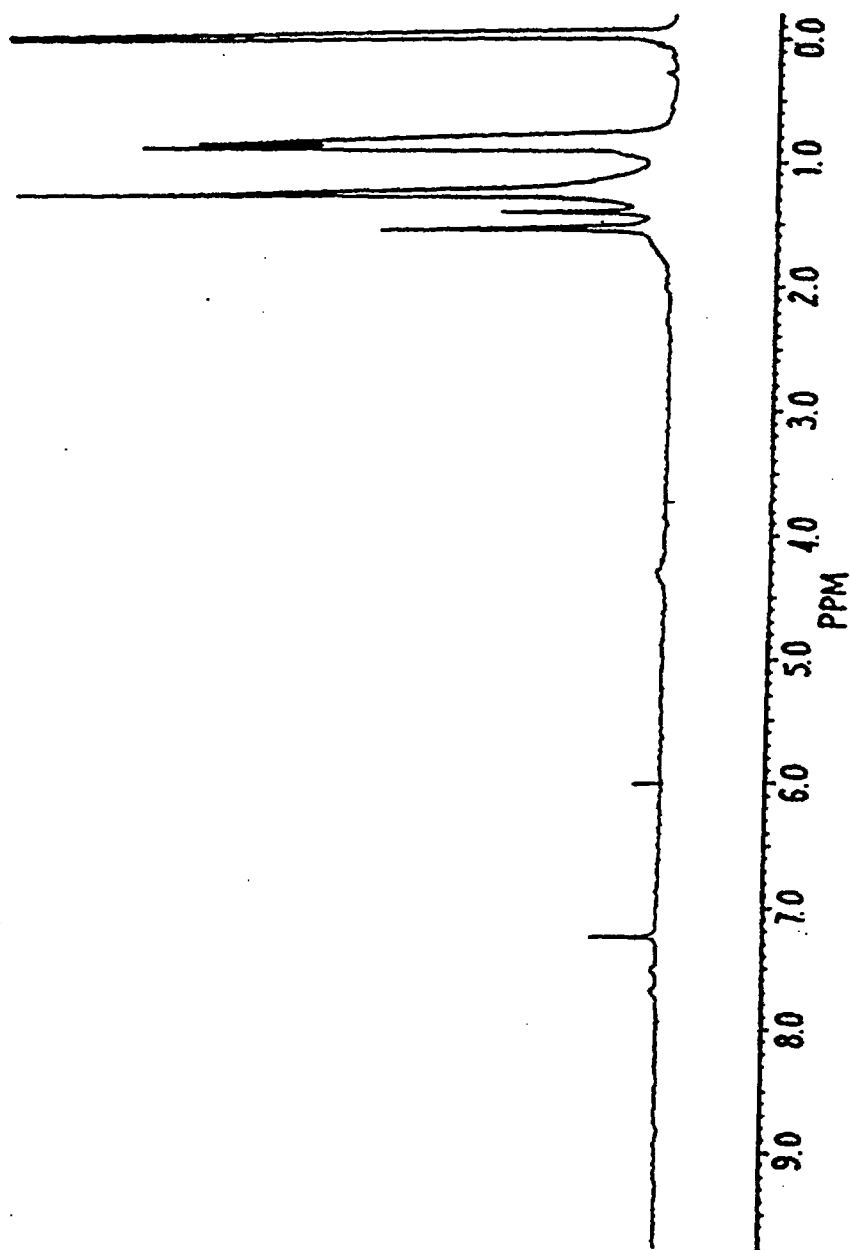


FIG. 16

REPLACEMENT SHEET

29 NOV 1997





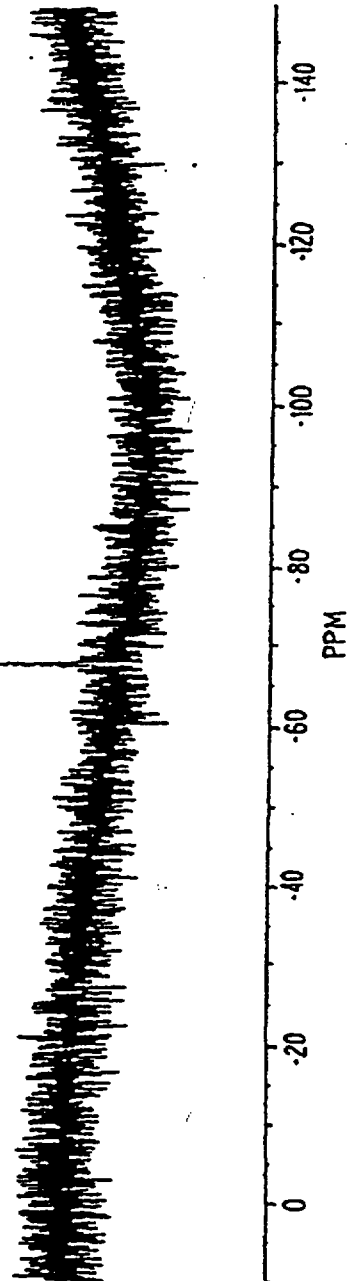
File 18

REPORT SHEET

29 NOV 1997

HERTZ

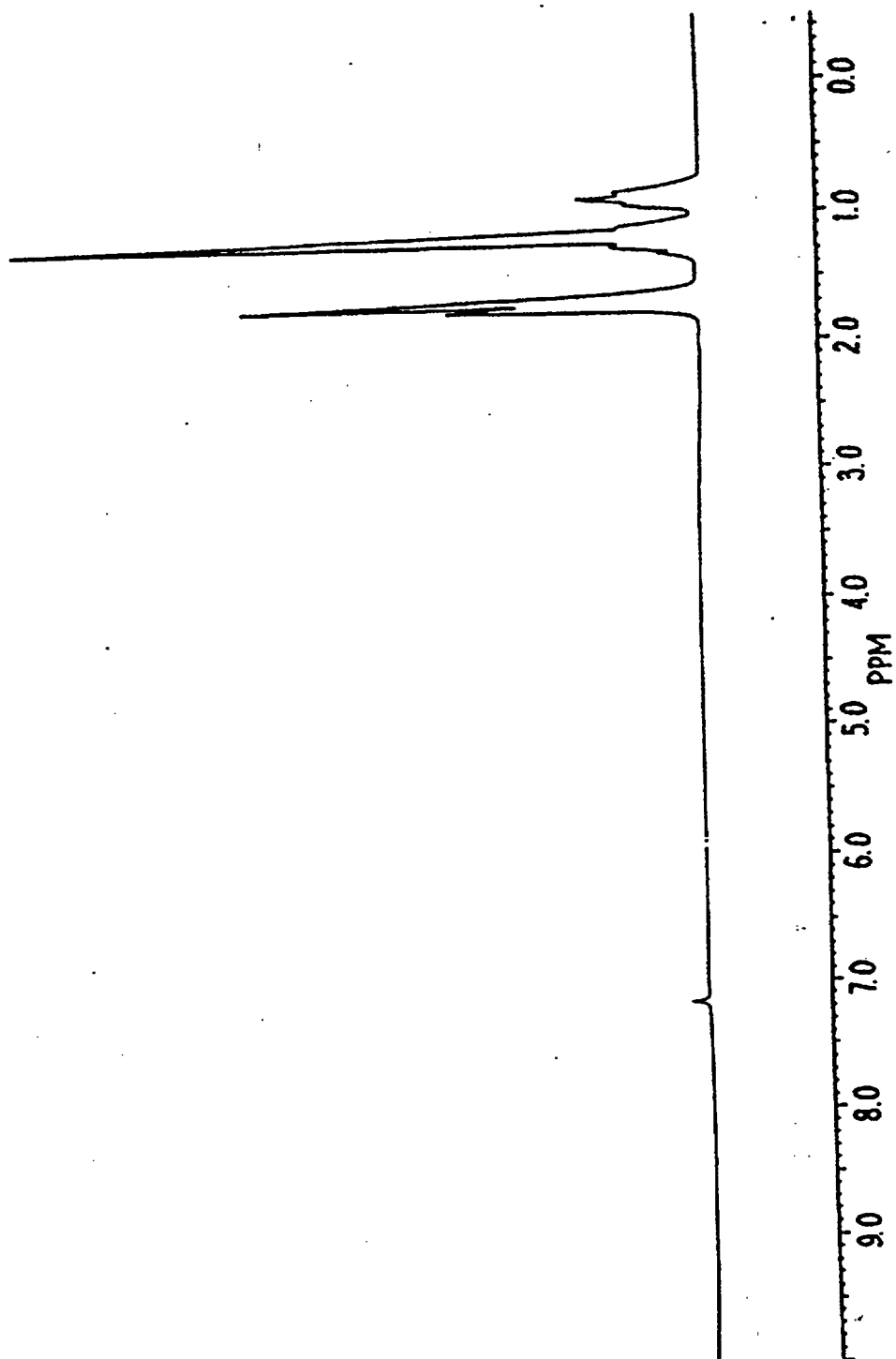
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Fig 19

REPLACEMENT SHEET

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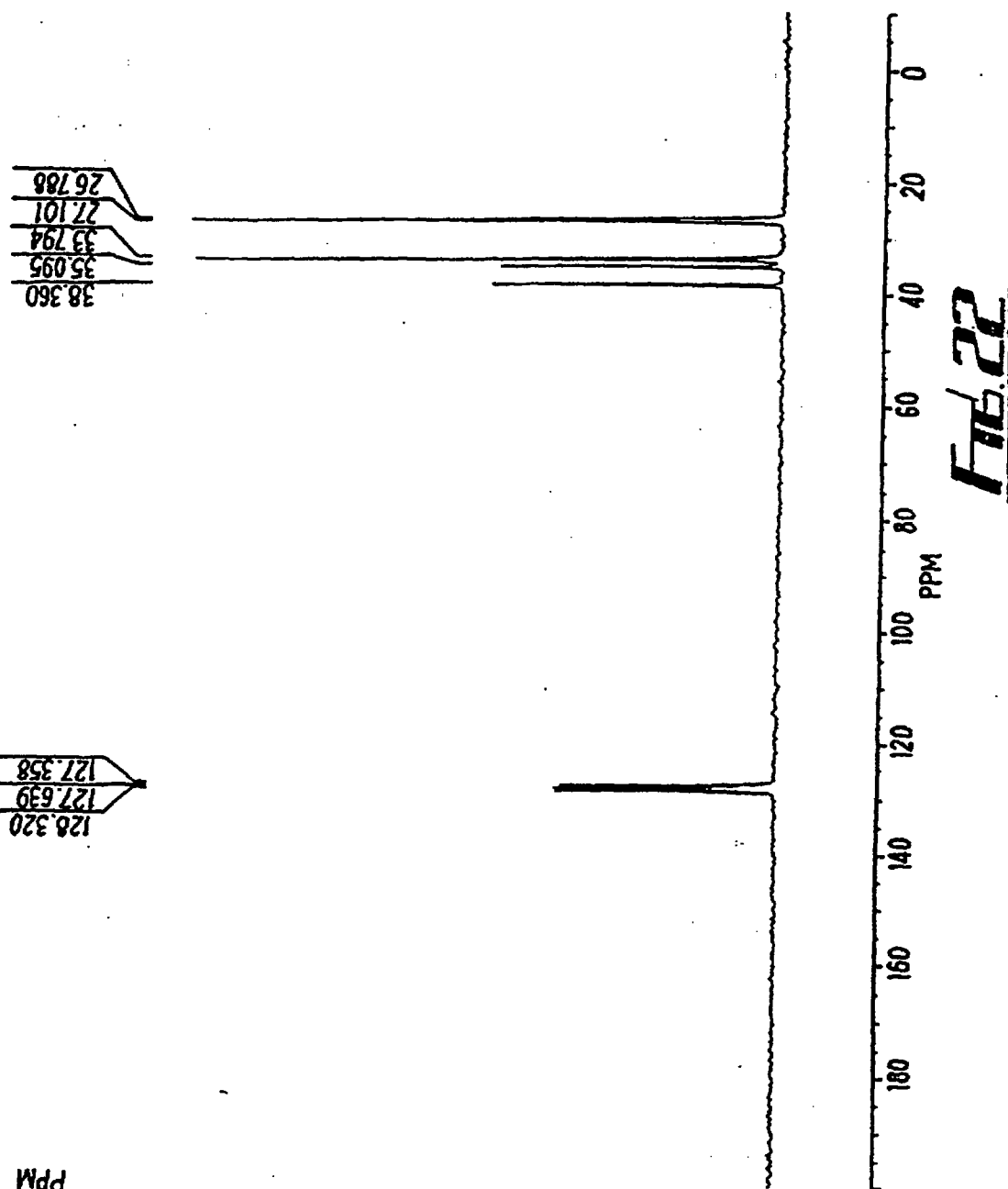
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File 21

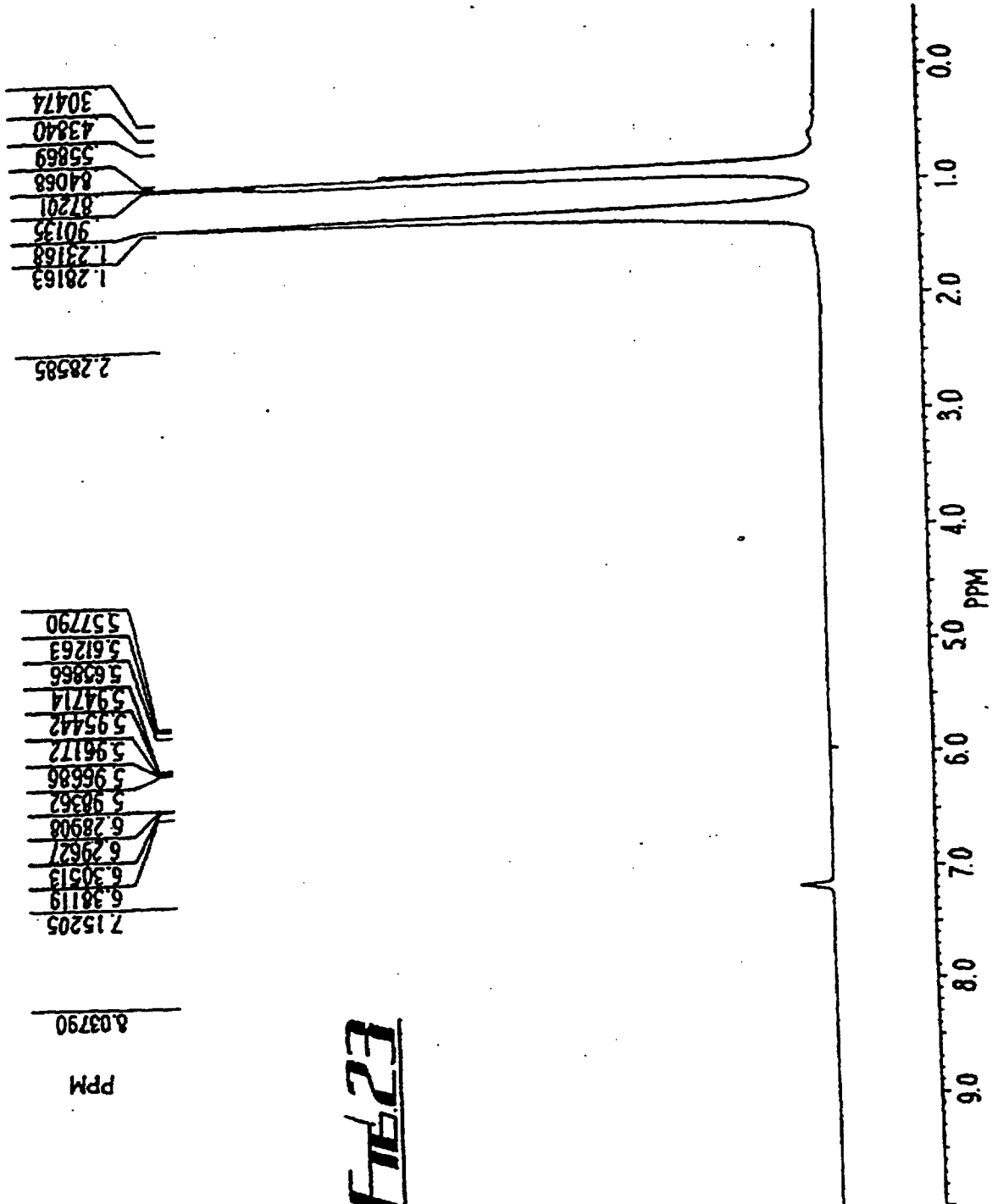
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REPLACEMENT SHEET

29 NOV 1997



File 23

REPLACEMENT SHEET

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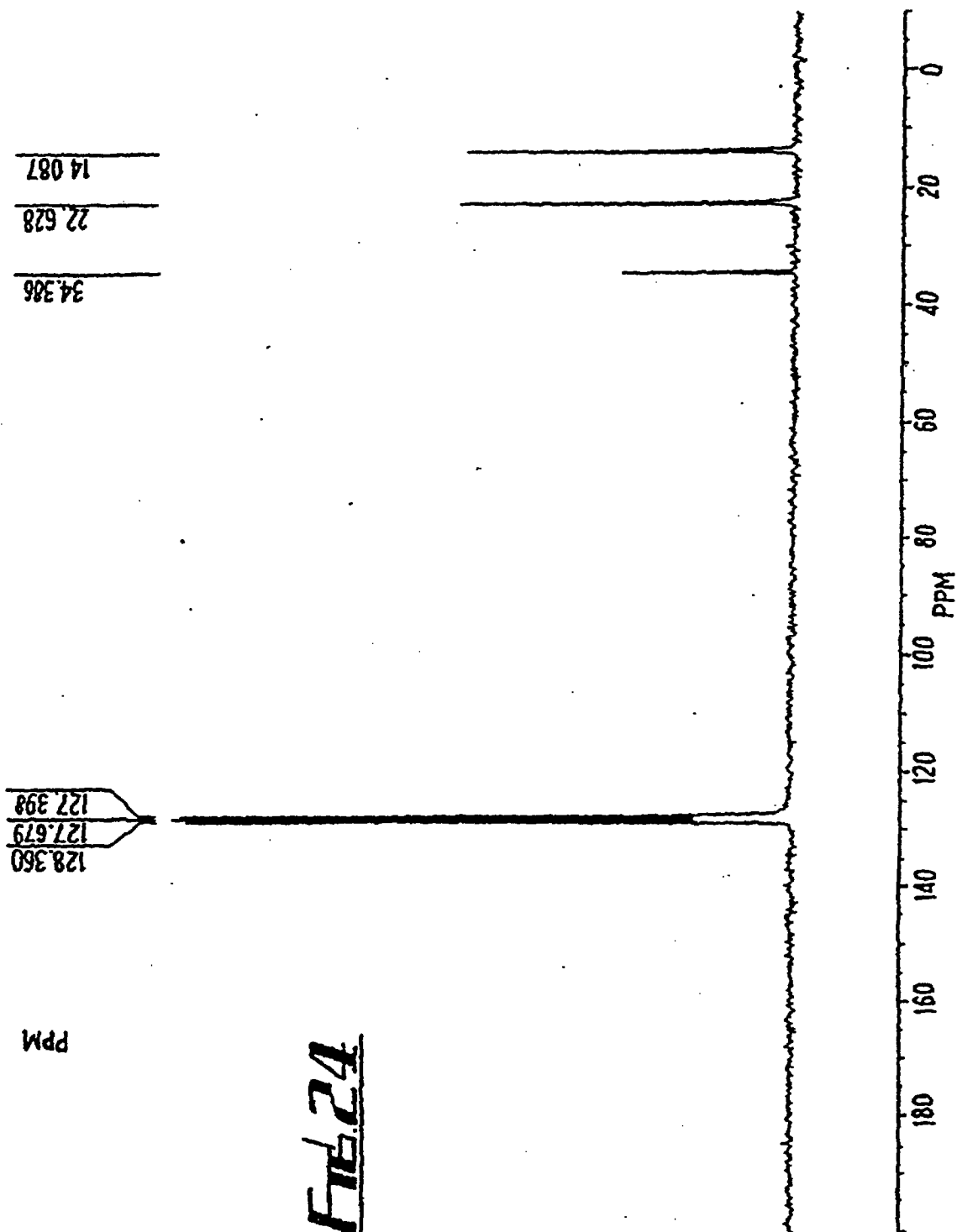


Fig. 24

REPLACEMENT SHEET

29 NOV 1997

-68.1337

Wdd

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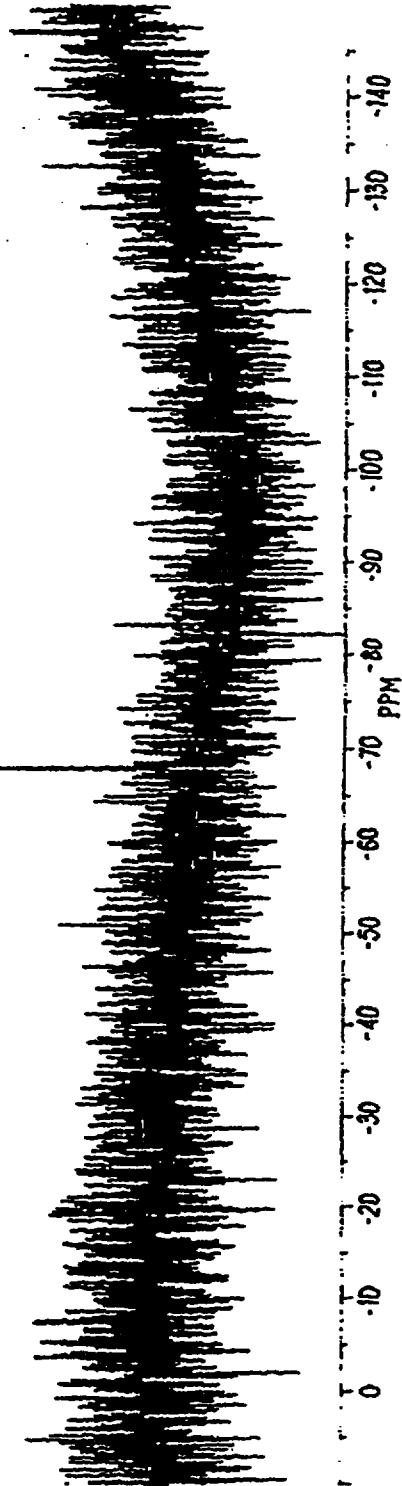


Fig. 25

1 ABSTRACT

2

3 METHOD OF DEPOSITION

4

5

6 There is described a method of chemically depositing a
7 substance. The method is of utility in the direct
8 manufacture of integrated circuits and in the
9 manufacture of a photomask for use in production of
10 integrated circuits. The method involves the use of a
11 compound which degrades into a deposit and a residue
12 when a radiant beam (eg a laser beam) or a particle
13 beam (eg an electron beam) is applied. The residue and
14 any unreacted compound may be washed off the substrate
15 to which it has been applied. Nanoscale dimensions of
16 the deposit can be achieved. A particularly suitable
17 organometallic compound is tetra-sec butyl diaurum
18 difluoride.

19

20

21

22 Representative drawing - Figure 2

23

24

25

26

REPLACEMENT SHEET

3 1 AUG 1999