From *Light Metals 1981*, Gordon M. Bell, Editor

BOILING POINT RISE OF BAYER PLANT LIQUORS

Light Metals

John L. Dewey

Alumina Research Division Reynolds Metals Company Bauxite, Arkansas

This paper presents a method for calculating the boiling point rise of Bayer plant caustic-aluminate liquors within about ± 0.1 °C from the chemical analyses of the liquors. The effect of impurities present in significant amounts is included.

The boiling point rise of these liquors has been found to be identical to the boiling point rise of pure sodium hydroxide solutions at equal molality and liquor temperature. This is consistent with the Debye-Huckel definition of total ionic strength for uni-univalent ions, indicating that divalent anions (CO_3^{--}, SO_4^{--}) may only be singly ionized in the concentrated solutions. The existence of AlO_2^{--} rather than $Al(OH)_4^{--}$ also is supported.

Principal sources of error are thought to be the chemical analyses, identification of organic constituents, and literature data on the vapor pressures of NaOH solutions.

Combination of the vapor pressure data with an equation of state for water permits calculation of the heat of evaporation.

Introduction

Knowledge of the boiling point rise (BPR) of Bayer plant streams is required for the design of evaporators and heat recovery units for new plants and for monitoring the operating efficiency after commissioning. Presently, BPR values must be determined experimentally, and values for use in the design of new plants must be estimated from prior experience.

Further, a portion of the economic benefits of operating expensive processes for control of impurity levels in plant streams derive from the savings of plant energy requirements that may be obtained by preventing the BPR values from increasing during the operating life of the plant. The value of operating these processes must be taken on faith since several years may be required to obtain a significant change in the composition of the large inventory of plant liquor.

A method of estimating BPR values from liquor compositions was sought as a means of improving the estimates of BPR for new plants and for identifying the individual and combined effects of various impurities found in Reynolds' Bayer plant liquor streams. An approach based on the work of Professor H. P. Meissner at Massachusetts Institute of Technology was chosen. The results obtained have been better than expected.

Presentation of Results

Figure 1 shows the agreement over a range of temperatures and molalities between the boiling point rise of Bayer liquors and of sodium hydroxide solutions at equal molalities for a high-temperature Bayer plant using 240°C digestion. The curves show the variation of the BPR of sodium hydroxide solutions with temperature at NaOH molalities equal to the molalities of liquors 20 to 29 of Tables I and II. The data points were obtained by Carithers and Brown (1) for 0.35 and 0.64 alumina to free soda (expressed as Na₂CO₃) weight ratios, for a range of free soda concentrations from 100 to 200 grams/kilogram liquor obtained by dilution with water. The liquor molalities (Table 2) range between 2.87 and 7.56, hence the liquors must be considered concentrated solutions that would not be expected to obey the classical laws of dilute solutions. The fit of the curves to the data points is thought to be satisfactory for Bayer plant use.

Tables I and II also present chemical analyses of 19 other liquors and the boiling point rise at 1 atmosphere pressure from a study by Sanders (2). In Table II "m" is the sum of the individual molalities of the anions shown (cations were considered in calculation of the water), "m¹" is the molality of the sodium hydroxide solution having the same boiling point rise as the liquor (equal water activity) and the "calc. BPR" is the boiling point rise of a sodium hydroxide solution of the same molality as the liquor. The difference between the observed and calculated BPR values is satisfactorily small and the standard error for the 29 liquors is only 0.14°C.

24	240°C Digestion 16 17 18 19 20 21 22 23	$(7) + Na_2 SO_4$ 12 Desalted 13 14	4 (4) +Na ₂ SO ₄ 5 6 143°C Digestion 7 8 9 10	Sinter Plant 1 2 3	Liquor Source No.
. 64 . 64	យយយយយយយយយយ ប្រទាំមួយ ដែលមួយ យោយយោ ប្រទាំមួយ ហើយ ហើយ ហើយ ហើយ ហើយ ហើយ ហើយ ហើយ ហើយ ហើ	. 372 . 372 . 377 . 377 . 377 . 377	.282 .282 .282 .372 .372 .372 .372	.271 .271 .271	Table A/C
8223 8223 823	.856 .823 .823 .823	.861 .861 .861	.908 .908 .677 .677	.934 .934 .934	<u>I. Ch</u>
100 125 150 175 200	173.87 166.32 154.73 147.12 100 125 125 125 125 125	153.57 200.27 170.23 148.18	144.12 142.04 188.09 155.84 149.85 144.29 138.63	158.14 150.06 142.41	emical Ana
121.52 151.90 182.28 212.66 243.04	203.10 194.28 180.75 171.85 121.52 151.90 182.28 212.66 243.04	226.85 226.85 232.52 197.64 172.04	158.71 156.40 207.13 230.21 221.36 221.36 213.15 204.79	169.35 160.69 152.50	alyses of
63.96 79.94 95.93 111.92 127.91	68.68 61.09 58.08 34.96 43.96 52.49 61.18 69.91	57.12 75.56 64.23 55.91	40.60 40.01 52.99 57.97 55.74 53.68 51.57	42.81 40.62 38.55	BPR-Stu Gra A
7.92 9.90 11.88 13.87 15.85	20.00 19.13 17.80 16.92 7.92 9.90 11.88 11.88 11.88 13.87	5.00 4.12 3.50 3.05	4445 000 44.80 050 051 051 051 051 051 051 051 051 05	000	ums/Kg Lio TOS
2.37 2.96 4.15 4.74	4.12 3.67 2.349 4.15 56 4.15	8.46 9.62 7.18	5.14 6.71 8.58 7.94 2.63	5.08	quor K20
4.97 6.22 7.46 9.95	223.30 222.29 20.74 19.72 4.97 6.22 7.46 8.70 9.95	2.43 2.47 2.47 2.10 1.83	0.59 0.58 0.77 2.47 2.38 2.29 2.29	0.64 0.58	<u>C1</u>
		1.53 0.79 0.67 0.58	0.39 0.38 0.51 1.55 1.49 1.44 1.44	0.43 0.41 0.39	
6.53 8.16 9.79 11.42 13.05	17.55 16.94 14.85 6.53 8.16 9.79 11.42 13.05	17.88 2.18 1.85 1.61	16.41 26.37 21.42 8.38 8.06 7.45 7.45 8.18	18.01 17.08 16.22	S04



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	25 27 28 29	20 21 23 24	16 12 19	13 14 15	7 9 10 11 12	のらよらって	Liquor No.	
	0.774 1.024 1.304 1.620 1.981	1.424 1.866 2.355 2.890 3.489	2.821 2.643 2.390 2.230	3.124 2.521 2.115	2.472 2.344 2.228 2.113 2.472 2.471	2.674 2.506 2.351 2.364 2.365 3.312	.0H	
	1.528 2.020 2.573 3.200 3.909	0.811 1.063 1.343 1.645 1.987	1.961 1.836 1.660 1.548	2.012 1.623 1.361	1.555 1.475 1.402 1.330 1.555 1.555	1.045 0.980 0.919 0.977 0.977 0.978 1.370	Liquor (Al02	
	0.171 0.226 0.287 0.357 0.437	0.166 0.217 0.274 0.336 0.406	0.955 0.895 0.809 0.755	0.095 0.076 0.064	0.096 0.091 0.086 0.082 0.675 0.675	0.023 0.021 0.020 0.020 0.020 0.020 0.029	C1	
	00000	00000	0000	0.057 0.045 0.038	0.112 0.106 0.101 0.096 0.112 0.112	0.028 0.027 0.025 0.025 0.025 0.025 0.025	n - Molal F	
	0.248 0.327 0.416 0.418 0.418 0.632	0.241 0.315 0.398 0.488 0.588	0.402 0.377 0.341 0.318	0.413 0.334 0.280	0.960 0.910 0.865 0.820 0.959	0.132 0.123 0.115 0.169 0.169 0.169 0.237	Basis - NaCO _s	
	0.083 0.110 0.140 0.174 0.212	0.0816 0.106 0.133 0.164 0.197	0.266 0.252 0.226 0.211	0.031 0.025 0.021	0.120 0.114 0.108 0.102 0.120 0.258	0.234 0.219 0.205 0.210 0.210 0.342 0.342	Anions or NaSO4	
	0.152 0.201 0.256 0.317 0.388	0.148 0.194 0.244 0.300 0.361	0.458 0.433 0.387 0.362	0.053 0.043 0.036	0.066	000000	ly Shown Org.	
	2.958 3.907 4.975 6.183 7.559	2.869 3.759 4.738 5.822 7.029	6.433 5.812 5.423	5.782 4.665 3.915	5.379 5.100 4.848 4.598 5.959 5.516	4.135 3.875 3.636 3.767 3.900 5.276	Total "m"	
	3.28 4.56 5.78 2.78 9.67	2.94 4.11 7.00 9.00	8.50 7.86 6.87 6.29	6.96 5.23 4.261	6.24 5.46 6.96 37	4.55 4.22 4.03 4.18 6.01	Obs. BPR (<u>1 atm</u>)	
Mean Std.	3.23 4.21 5.07 7.52	2.85 3.87 5.85 7.10	6.80 5.76 5.39	5.81 4.67 3.97	5.37 4.83 5.40 5.42	4.18 3.95 3.70 3.80 3.93 5.20	m ¹ From Obs. BPR	
Dev.	2.97 4.17 7.55 9.78	2.87 3.97 5.33 6.97 8.93	8.61 7.95 6.95	6.90 5.23 4.16	6.30 5.87 5.50 5.10 6.50	4.45 3.80 3.96 6.10	Calc. BPR (from m)	
0.02	0.31 0.39 0.09 0.23 -0.13	0.07 0.14 0.03 0.03	-0.11 -0.09 -0.08	0.06	0.06 0.01 0.04 0.02 -0.22 -0.23	0.10 0.10 0.08 0.07 0.04 -0.09	∆ BPR Obs Calc.	
1.00 ⁸	1.093 1.078 1.019 1.027 1.027 0.995	0.993 1.029 1.002 1.005 1.010	0.991 0.993 0.991 0.994	1.005 1.001 1.014	0.998 1.000 0.996 1.000 0.977 0.977	1.011 1.019 1.018 1.009 1.008 0.986	Ratio m ¹ /m	

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Table III shows the results of calculations made to determine the most useful method of expressing the composition of the liquors. The comparative results are expressed as the value of m^1 divided by the Debye-Huckel ionic strength of the solution calculated from the data of Table II by three different assumptions, viz:

- 1. (Column 3) Uni-univalent ions as shown in Table II.
- (Column 5) Full dissociation to a mixture of univalent cations and uni- and di-valent anions; and
- (Column 7) Uni-univalent ions with Al(OH)₄instead of Al0₂-.

It is apparent that the assumption of uni-univalent ions is more useful than the assumption of mixed valency for the concentrated solutions normally encountered in a Bayer plant. (This might not be true for very dilute liquors.) Use of the tetrahydroxy ion yields a ratio different from unity that also shows a statistically significant variation with the solution concentration. It is believed that this comparison supports the existence of AlO_2^- in the concentrated liquors.

Derived Data and Assumptions

Computer Algorithm of Boiling Point Rise

Handbook data (3) for the vapor pressure of water over sodium hydroxide solutions over the ranges 0 to 10 molal and 60 to 300°C were converted to boiling point rise values and correlated with the aid of a stepwise multiple regression computer program. The resulting equation is:

BPR (°C) = $0.00182 + 0.55379 (M/10.)^7 + 0.0040625MT +$ (1/T) (-286.66M + 29.919M² + 0.6228M³) - $0.032647M (MT/1000.)^2 +$ (T/1000.)⁵ (5.9705M - 0.57532M² + 0.10417M³) (1) Std. error of estimate = 0.068°C M = Molality - gm. mols/kg H₂0 T = K = °C + 273.150

The constant may be omitted.

Equation 1 is believed to represent the boiling point rise of sodium hydroxide solutions within the accuracy of the available data and therefore the boiling point rise of Bayer liquors with comparable accuracy.

Table

H

Reduced

Data

from

Boiling

Point

Rise

Studies

Table III. Test of Correlation Methods

Liquor No.	Univa Anio m ¹	lent ns m1/m	Uni + d 	ivalent ons <u>m¹/I</u>	Univalen With Al <u>m</u> 2	t Anions (OH) ₄ <u>m¹/m₂</u>
No. 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25	m ¹ 4.18 3.95 3.70 3.80 3.93 5.20 5.37 5.10 4.83 4.60 5.42 5.42 5.81 4.67 3.97 6.80 6.39 5.39 2.85 3.87 4.75 5.85 7.10	m ¹ /m 1.011 1.019 1.018 1.009 1.008 0.998 1.000 0.995 1.000 0.995 1.000 0.995 1.000 1.014 0.993 1.029 1.029 1.022 1.005 1.010 1.010	I 4.867 4.559 4.525 4.922 6.343 7.700 7.297 6.935 6.585 8.282 8.113 6.788 5.475 4.593 8.525 8.014 7.226 6.740 3.621 4.744 5.982 7.354 8.879 3.726	m^1/I 0.859 0.866 0.865 0.840 0.799 0.697 0.699 0.697 0.699 0.697 0.699 0.697 0.699 0.703 0.668 0.853 0.864 0.798 0.797 0.800 0.787 0.816 0.794 0.796 0.800 0.867	m_2 4.297 4.017 3.760 3.903 4.042 5.555 5.721 5.407 5.125 4.846 6.339 5.866 6.245 4.961 4.121 7.391 6.900 6.190 5.751 2.960 3.915 4.990 6.206 7.595 3.130	m^{1}/m_{2} 0.973 0.983 0.984 0.974 0.972 0.936 0.943 0.942 0.949 0.855 0.924 0.930 0.924 0.930 0.924 0.931 0.937 0.963 0.989 0.952 0.943 0.923 1.032
26 27 28 29 Mean S.D.	4.21 5.07 6.35 7.52	1.078 1.019 1.027 0.995 1.008 0.025	4.927 6.276 7.804 9.544	0.855 0.808 0.814 0.788 0.797 0.060	4.219 5.494 7.005 8.825	0.998 0.923 0.906 0.852 0.945 0.037

Heat of Vaporization of Bayer Liquors

The heat of vaporization of Bayer liquors may be calculated by rigorous thermodynamic relations (4) with the aid of an equation of state for water or the steam tables, using Equation 1 and water vapor pressures to calculate $\Delta p / \Delta T$ for use in the standard relation

$$\Delta H_{V} = (\Delta p / \Delta T) (T, M) (V_{V} - \overline{V})$$
(2)

 $\triangle Hv =$ heat of vaporization, in PV units.

- V_V = vapor volume at the solution temperature and the water saturation pressure at (T-BPR).
- \overline{V} = partial molar volume of liquid H₂0 in the solution at T.

It is assumed that \overline{V} at T can be taken as the partial molar volume of H_20 at 20°C times the ratio of the volume of liquid water at t°C to that at 20°C. The relation

$$\label{eq:relation} \begin{split} 1/\bar{v} & (20\,^\circ\text{C}) = 1.042753 + 0.045431 \text{sin} \; ((\text{M-7.4})/5.9) \quad (3) \\ \text{std. error of } \bar{v} &= 0.049\% \; \text{of } \bar{v} \\ \bar{v} & \text{in } \text{cm}^3/\text{gram } \text{H}_20 \\ \text{M} &= \text{Molality} \end{split}$$

The argument of the Sin is in radians.

for sodium hydroxide solutions at $20 \,^{\circ}$ C, and thus also for Bayer liquors of equal molality, was devised from published data (5) on the density of sodium hydroxide solutions.

Calculated values of boiling point rise and heat of vaporization for a range of molalities and temperatures are tabulated in Table IV. Values are given to 3 and 4 significant digits, respectively, for convenience of those wishing to prepare graphs.

Definition of Organates

No rigorous definition of the sodium organate compounds present in the liquors has been found. The following assumptions, discussed in Appendix 2, were used in the present study and appear to be satisfactory.

- A. For 140°C digestion plant
 - 100% sodium oxalate, molecular weight 134, two acid groups per molecule.

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NOTES: E	」 、 、 、 、 、 、 、 、 、 、 、 、 、	Molality	Solution Temp., °C	Tak
)PR: N: Malality:	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	BPR	ບ ຫ	ble IV.
Boil Heat gm m	2382 2375 2375 2376 2376 2384 2398 22418 22418 22418 22418 22474 2574	HV	0	BPR an
ing poin of Vap ols/Kg 1	0.86 1.87 3.00 4.24 5.59 7.04 8.58 10.22 11.99 13.94	BPR	10	ld Heat
nt ris orizat H ₂ 0	$\begin{array}{r} 2225\\ 22255\\ 22255\\ 22258\\ 22263\\ 22273\\ 22326\\ 232$	HV	0	of Vap
e, °C ion, Kj,	0 1.18 2.47 3.85 5.31 6.82 6.82 9.97 11.61 13.30 15.11	BPR	15	orizati
/Kg va	2115 2119 2125 2125 2133 2145 2145 2176 2176 2218 22196 2218 2218 22177	HV	o	on of
por	0 1.50 4.73 6.40 8.09 9.79 11.47 12.47 14.84 14.84	BPR	20	Bayer L
	1940 1949 1960 1972 1972 2003 2003 2021 2040 2060 2060 2083	HV	0	iquors
	0 1.84 3.73 5.65 7.57 9.47 11.34 11.34 12.17 14.95 16.70 18.47	BPR	25	and Na
	1713 1730 1748 1768 1768 1787 1807 1807 1828 1848 1848 1868 1868 1889	HV	0	0H Sol
	0 2.21 4.44 6.66 8.85 11.01 13.11 15.15 17.15 17.15 17.15 17.15	BPR	30	utions
	1404 1437 1437 1500 1530 1559 1559 1585 1611 1611 1634 1656 1677	HV	0	

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B. For 240°C digestion plant 20 mol % sodium oxalate, 45 mol % sodium acetate, 35 mol % sodium formate, Mean molecular weight 87.5, 1.2 acid groups per mean molecule.

The mols of organate in solution are calculated from the total organic soda (TOS in Table 1), which is obtained as the difference in total titratable soda between the liquor and a sample of the liquor that has been dried and burned on 0_2 or air to destroy the organic materials.

Sources of Error

The major sources of error are believed to reside in the chemical analyses. Titration analyses by competent analysts on liquors and accurate dilutions of liquors made over a period of time (i.e. not simultaneous duplicates) have shown standard deviations about the mean values of 0.8% of the value for free soda (C), 0.6% for total titratable soda (S), 1.5% for alumina (A), and 4.4% for carbonate (S-C). Since these errors affect both the amount of the compound and the amount of water in opposite ways, the effects on the calculated molality values are greater. Thus it is recommended that mean values of a number of analyses taken over a period of time be used for boiling point rise computations.

The accuracy of the assumptions for organate ions can be assessed only from the fit of the molality-BPR correlation. For plants with substantially different digestion conditions than those cited or with very high organate levels it is recommended that the organate assumptions be tested against BPR data and revised if necessary.

The sodium hydroxide solution vapor pressure data and the correlation for BPR derived therefrom are believed to be sufficiently accurate to satisfy the needs of plants for BPR data. Calculation of the heat of vaporization requires obtaining the derivative dp/dt, or preferably $\Delta p/\Delta t$. This should introduce additional error but data for testing is limited. Thus the accuracy of the heat of vaporization values is unknown.

Appendix

Calculation of Molality from Chemical Assay

The objective is to obtain as accurate a value as possible of the total molality of the liquor. All compounds dissolved in the liquor should be considered, but in practice some of the trace impurities with the lowest concentrations may be neglected without introducing intolerable errors.

A sample calculation of the molality of liquor No. 16 is presented in Tables V and VI to illustrate the method. Table V presents the liquor analysis after converting all values to grams per kilogram liquor, and a division of the alkaline elements into sodium and potassium components (trace amounts of lithium in the

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Table V. Liquor No. 16

Liquor Analysis - in grams/Kg liquor¹

C (Free Soda as Na ₂ CO ₃	173.87
S (Titratable Soda as Na ₂ CO ₃)	203.10
A	68.68
Total Organic Soda (TOS) as Na ₂ CO ₃	20.00
K20	4.12
Cl	23.30
F	0.0
s0 ₄	17.55

Total Alkali Calculation

S	203.10
TOS	20.00
Cl x 105.99/(2x35.45)	34.83
$F \times 105.99/(2x19.0)$	0.0
S0 ₄ ⁼ x 105.99/96.07	19.36
Sum = Total alkali as Na ₂ CO ₃	277,29
Less K ₂ 0 x 105.99/94.20	4.636
Total Na20 as Na2CO3	272.65

¹Mean value of assays on original liquor and on three dilutions calculated back to original concentration.

Table VI. Liquor No. 16 - Molality Calculation

ion	Wgt. Calculation	gms ion per Kg Liq.	M.W. ion	Gram Per Kg _Liq.	Mols Per Kg H ₂ 0
OH AlO ₂ Cl F	2x17x(C/105.99-A/101.96) Ax2x58.98/101.96	32.873 79.458 23.30 0.0	17.0 58.98 35.45 19.0	1.9337 1.3472 0.6573 0.0	2.804 1.953 0.953
CO ₃	(S-C)x60.01/105.99	16.551 17.55	60.01 96.07	0.2758	0.400
ORG.	105x2x59.9/(1.2x105.96)	18.844	59.9	0.3146	0.456
Sum An	ions	188.576		4.7113	6.831
Na ⁺	Tot. Na ₂ CO ₃ x2x22.99/105.99	118.280	22.99		
К÷	K ₂ Ox2x39.10/94.20	3.420	39.10		
Total N	Weight ions, gms.	310.276			
Wgt. H	20 1000Wgt. ions	689.724			
Multip	lier = 1000./wgt. H ₂ 0			1.4499	
Molality for B.P.R. Calculation = 6.831					
	0				

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liquors were neglected). Table VI presents the calculation of the weights and molar amounts of the anions and cations, the weight of water by difference and the molality. The calculations are simplified if the anions are taken as completely dissociated (i.e., free of Na or K) and the total weight of Na and K calculated separately, as shown in Table VI. Numerical coefficients of the conversion equations are given in terms of molecular weights and ions per molecule to assist interpretation by the reader.

Development of Organate Assumptions

Diluted samples of a high-temperature and of a lowtemperature plant liquor were assayed for total organic soda (TOS) by ignition, oxidizable carbon by permanganate titration, and sodium oxalate and a molar balance was made as follows:

	High Temperature Liquor	Low Temperature Liquor
Mols COO ⁻ from TOS	0.2266	0.0362
Mols COO ⁻ in oxalate	<u>0.0587</u>	<u>0.0345</u>
Excess mols COO ⁻	0.1679	0.0017
Mols C by permanganate	0.1319	0.0836
Mols C in oxalate	<u>0.0587</u>	<u>0.0345</u>
Excess mols C	0.0732	0.0491
Ratio - Excess C/Excess COO	0.44	28.9

For the low-temperature liquor (143°C) the number of acid groups calculated from the oxalate assay is nearly equal to the total number of acid groups calculated from the total organic soda (TOS) assay. Thus the mols of carbon found by the permanganate titration in excess of those with the oxalate must represent carbon groups in a relatively minor amount of undecomposed, or partially decomposed, humic acids. Since the data indicated the presence of only minor molar amounts of other organates, it was thought that the best estimate of the organates would be obtained by assuming that the acid groups found by the TOS assay are all present as oxalates.

Results obtained for the high-temperature liquor are quite different. Acid groups with oxalate account for only 26% of the total acid groups calculated from the TOS assay. Also, the mols of carbon obtained by the permanganate assay are not sufficient even to account for the acid groups present, suggesting the presence of low molecular weight acids such as acetic acid that are not easily oxidized by permanganate. No data on the distribution of low molecular weight organates in hightemperature liquor was found. However, G. Lever (7) reported, for a low-temperature plant liquor, a molar distribution of 7% Succinic, 1% lactic, 17% oxalic, 45% acetic and 30% formic acids with a mean ionic weight of 65.4 and 1.24 acid groups per mean ion. For the higher temperature plant, a molar distribution of 20% oxalic, 45% acetic, 35% formic acids, mean ionic weight

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59.9 with 1.20 acid groups per mean ion, was assumed to allow for greater degradation during digestion. The assumption is arbitrary and may not be satisfactory for all plants.

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